JOURNAL OF

CHEMICAL EDUCATION

VOLUME 30 March 1953 NUMBER 3

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Fig. 1. Side view.

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Fig. 2. Rear view, showing spring opposed jaws.

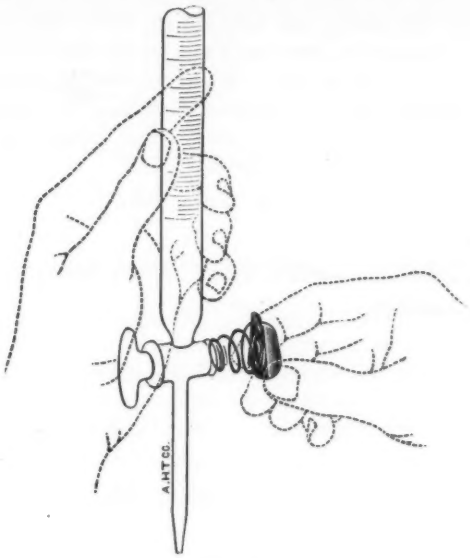

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Fig. 3. Showing method of attachment or removal.

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Fig. 4. In position.

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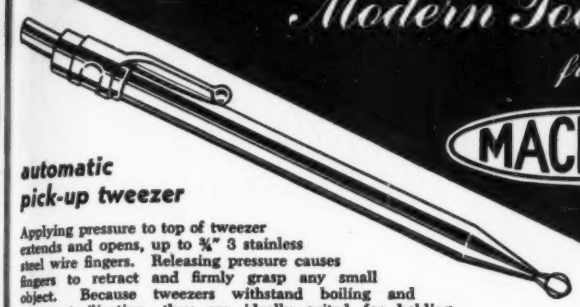

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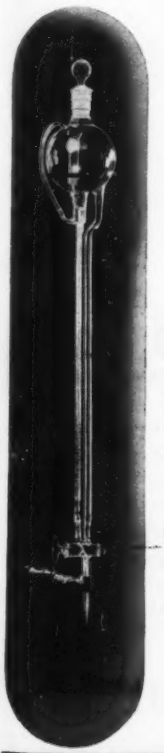

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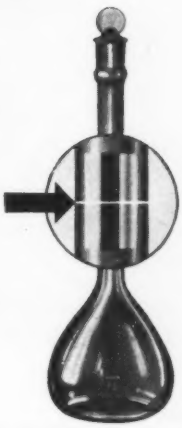

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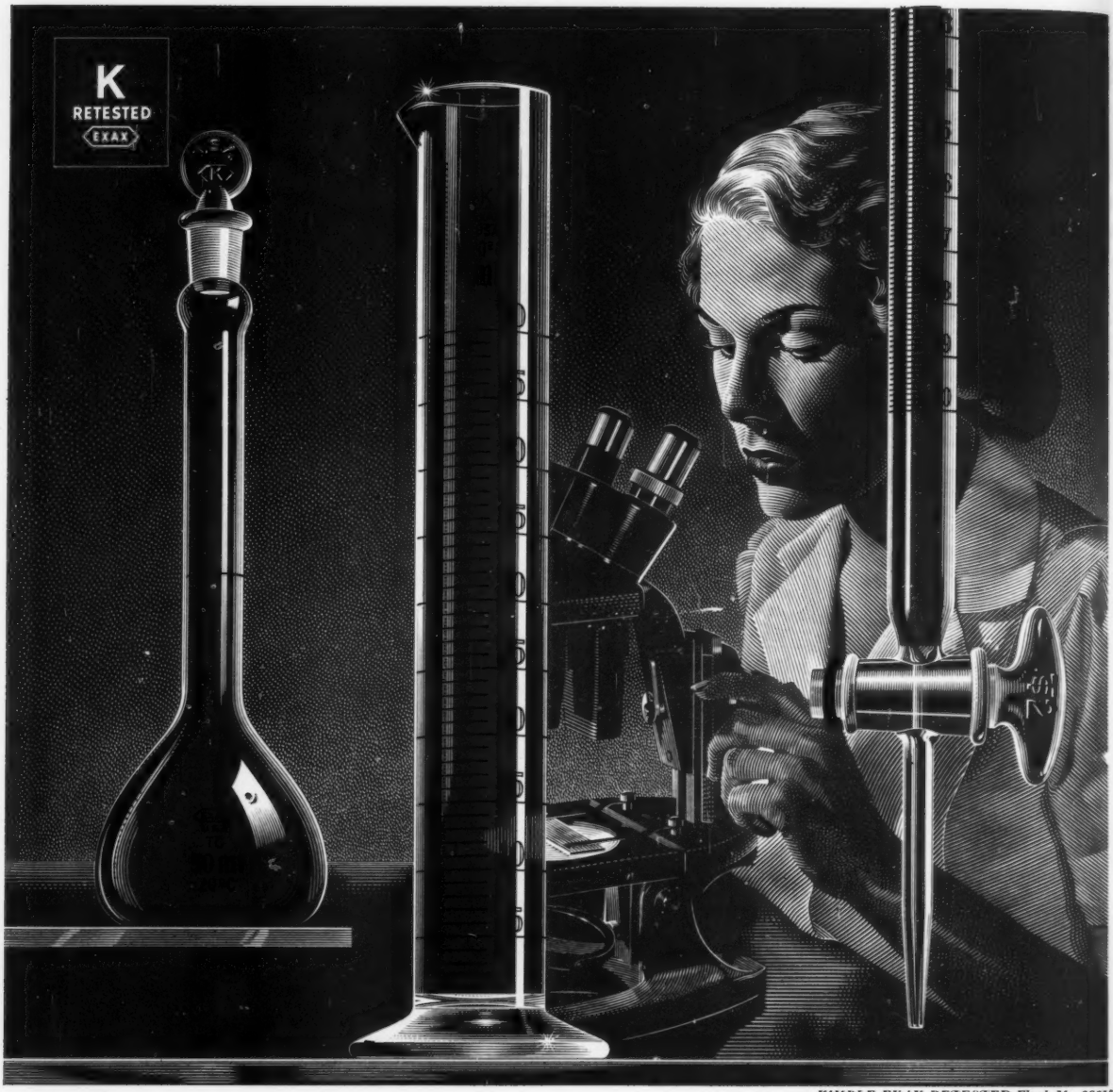

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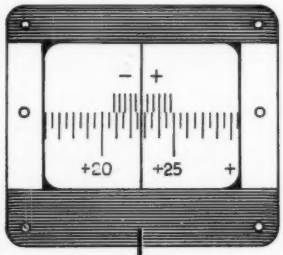

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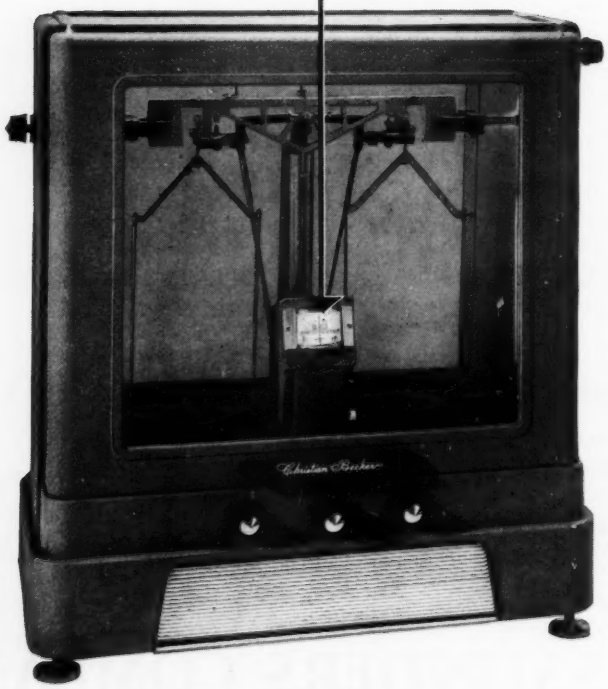

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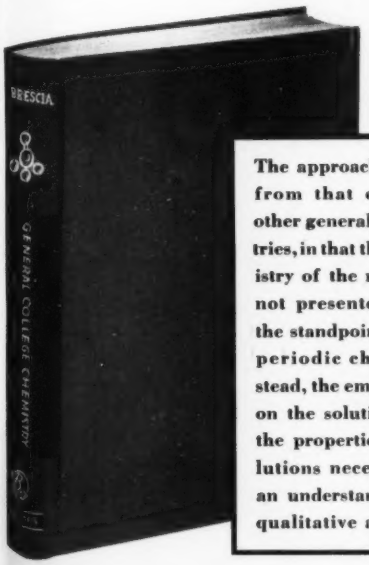

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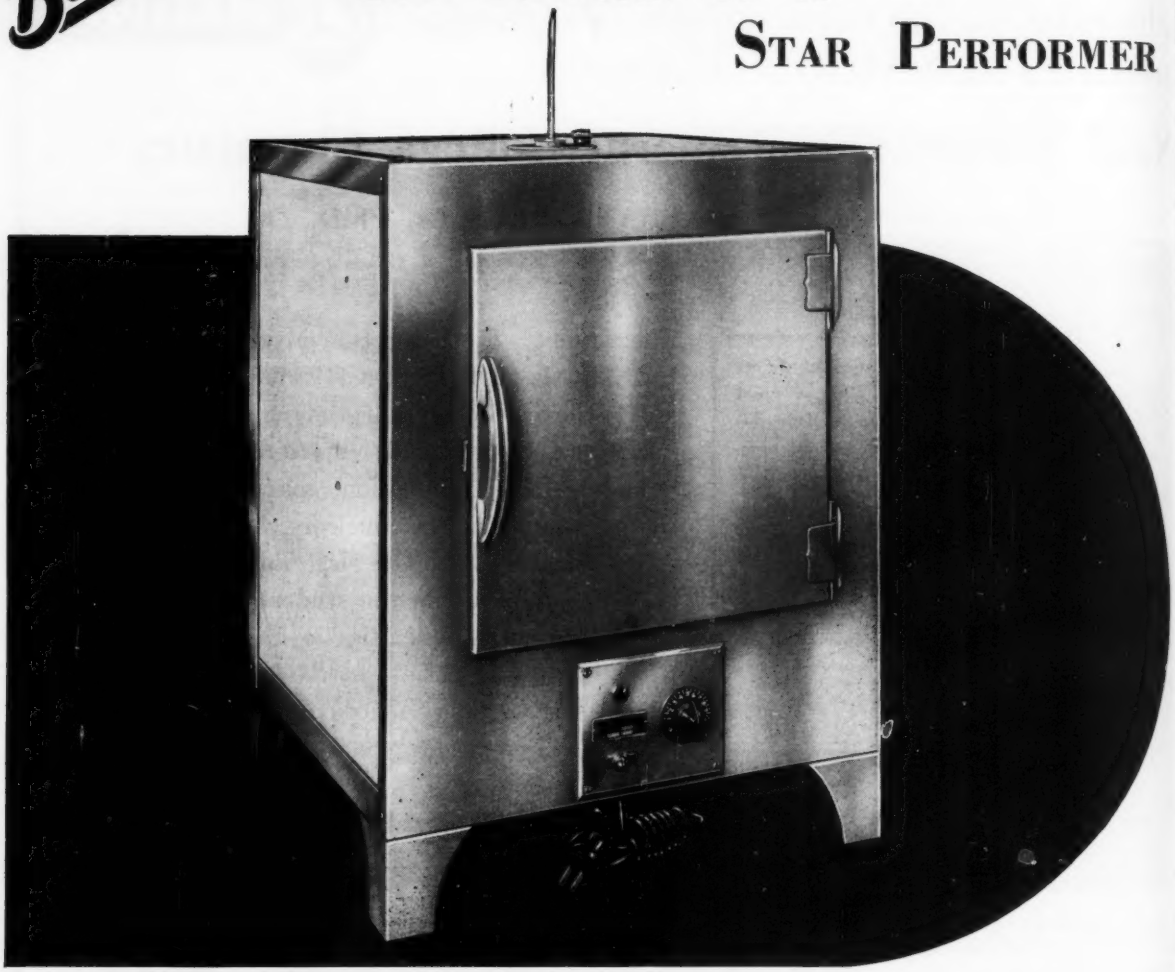

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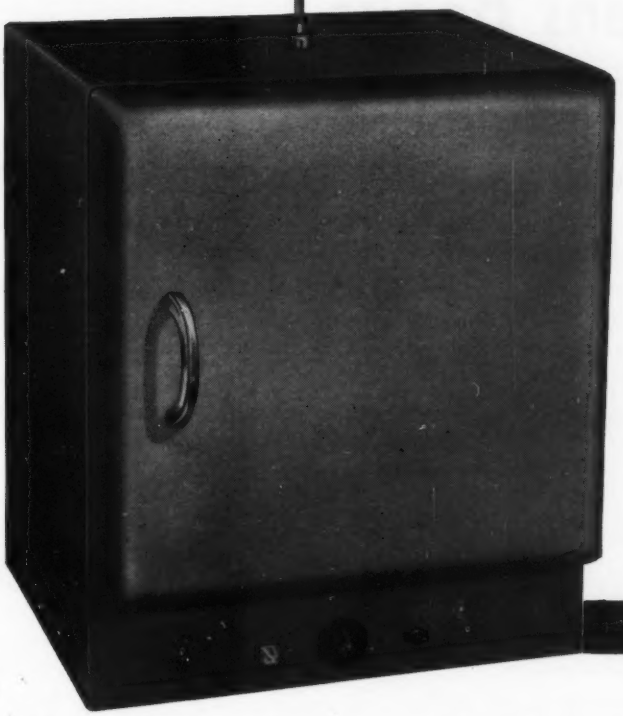

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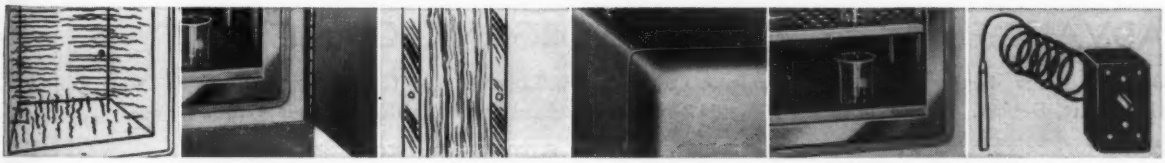

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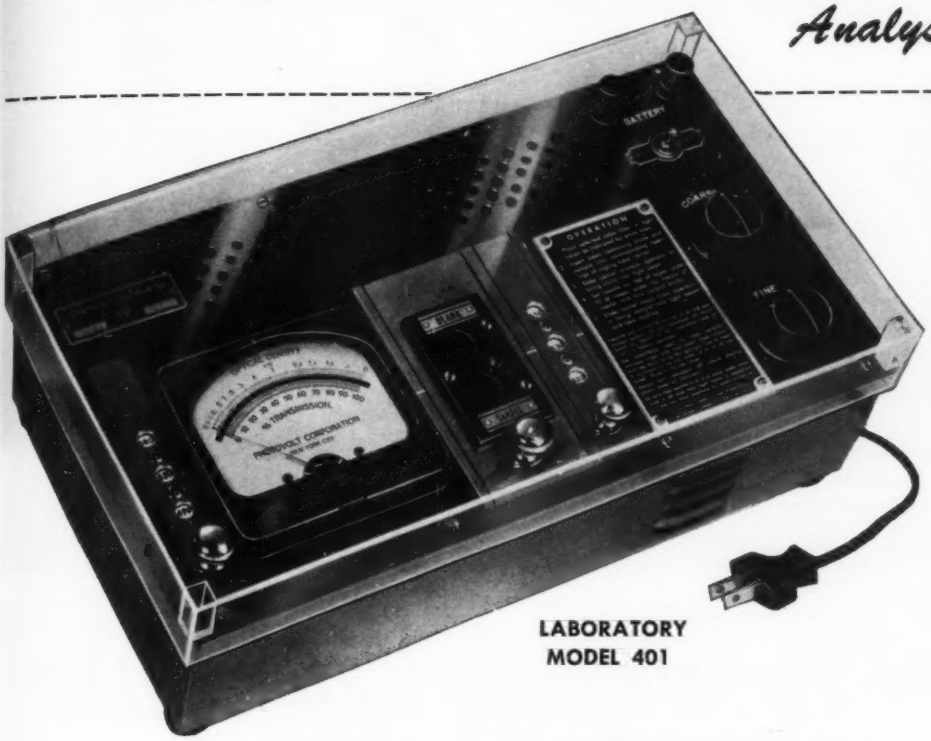

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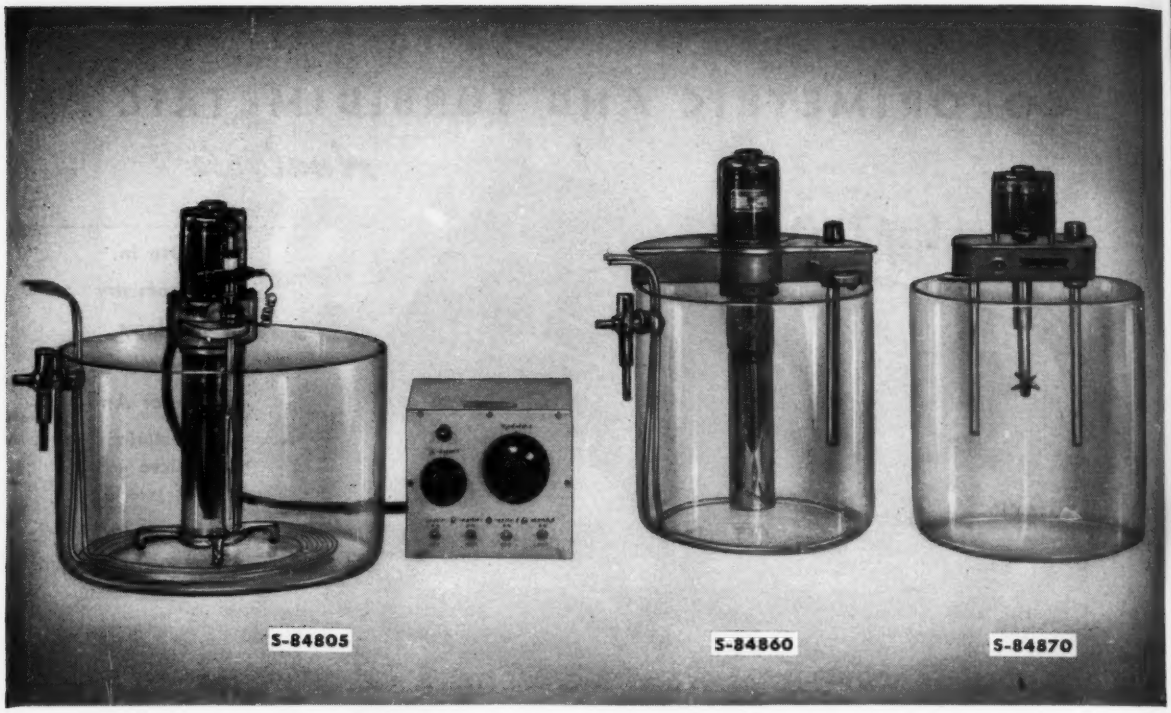

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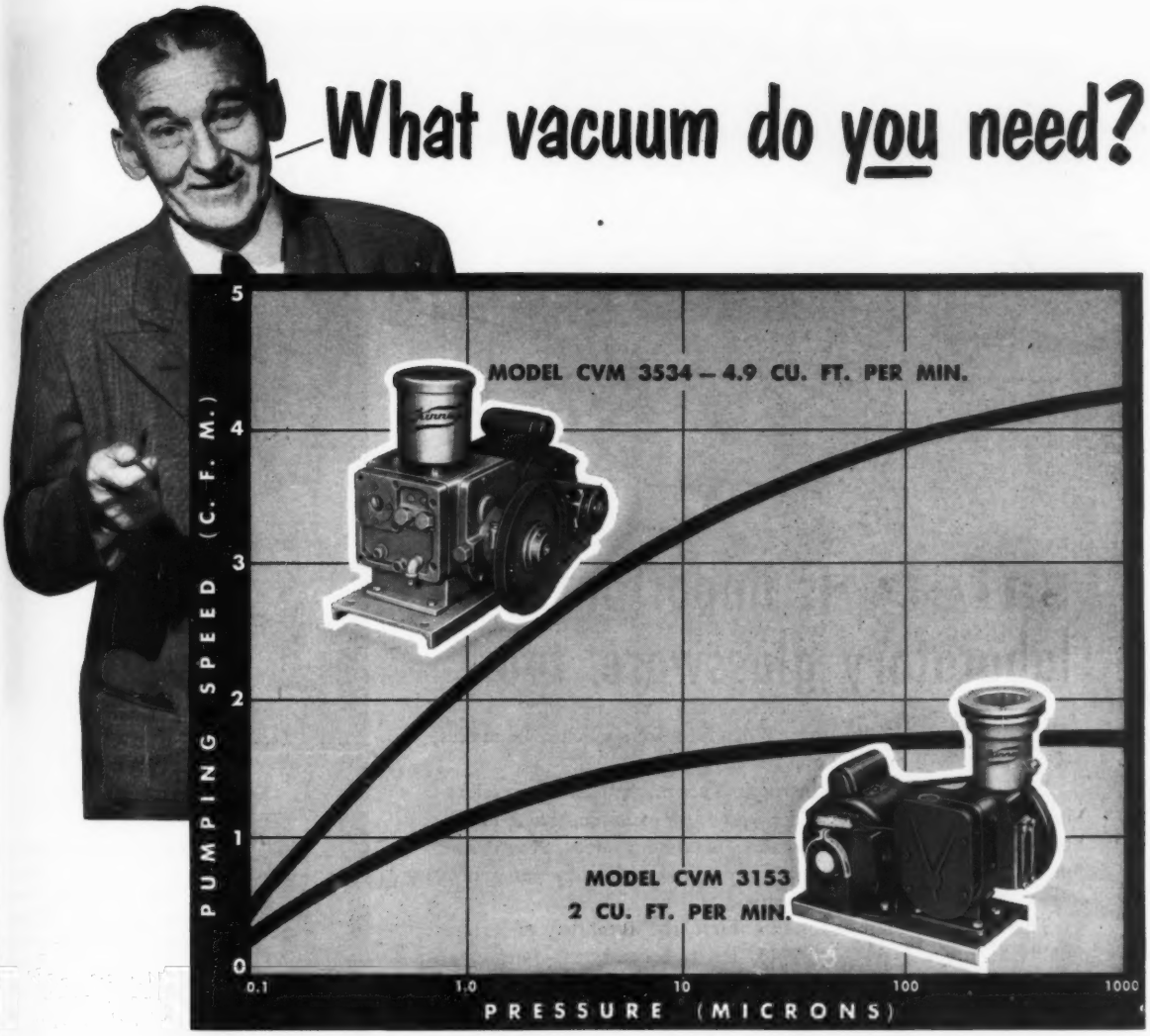

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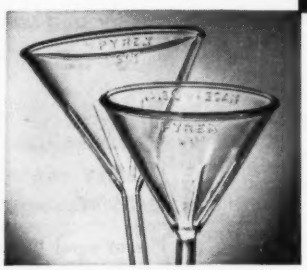

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Vladimir N. Ipatieff

(See page 110)

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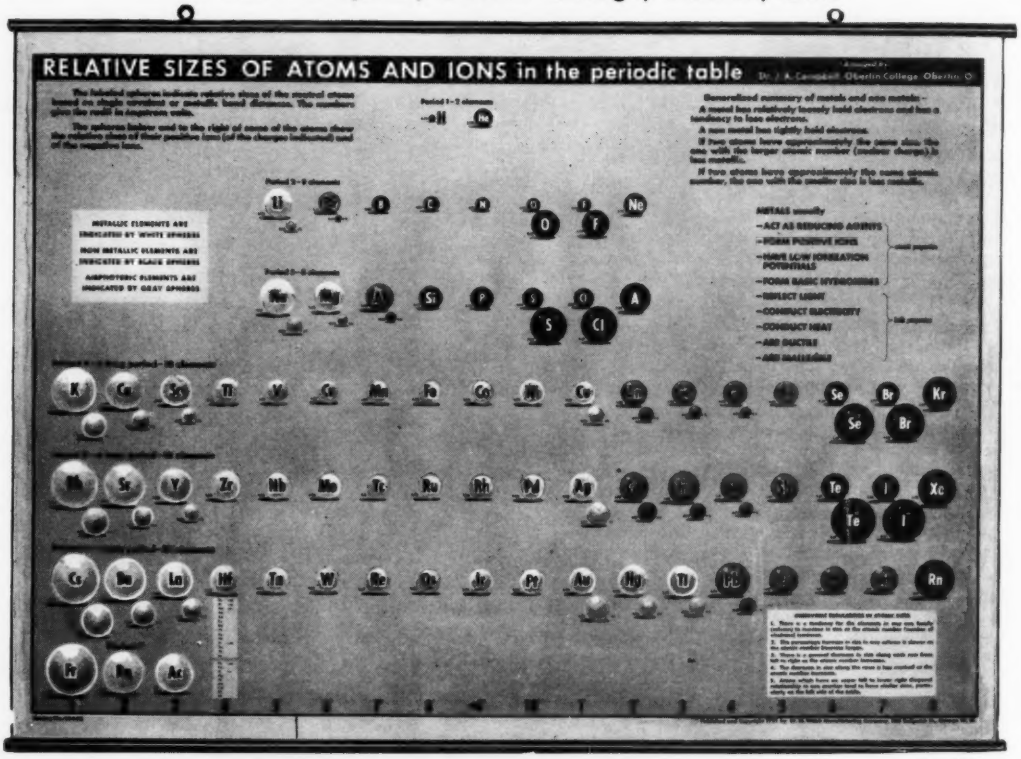

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Editor's Outlook

It is not often that I use this page for a book review, but in the present instance I cannot resist the temptation to do so. Although it was published in 1950, there has just come to my attention the book, "Effective Teaching," written by Fred C. Morris of Virginia Polytechnic Institute.[1] This little book of 86 pages was sponsored by the Committee on Teaching Manual, of the American Society for Engineering Education, and I believe is the best thing of its kind that has yet appeared.

Reviewing it is easy (or difficult) because one can open it to almost any page and find something which is well worth quoting and discussing. Although it was written particularly for the engineering instructor, every bit of it is just as appropriate for the teacher of chemistry. Every young teacher in any scientific field will profit from reading and digesting it.

Its principal thesis, of course, is that effective teaching is not always the result of an accident of birth but can also follow as a consequence of training, practice, and attention to the proper details. The effectiveness with which the author points out these details is a tribute to the title of the book and, no doubt, to his own accomplishments as a teacher.

In an early chapter he describes the ideal toward which to strive:

> When the desirable intellectual and personal characteristics of a teacher are enumerated, the list becomes so long that few people bother to finish reading it; they realize that there are no such teachers. To give just a thumbnail sketch of teacher qualifications, we might say this: A good teacher is one who has the sincerity of purpose that springs from sheer honesty of heart; the intellectual capacity and the energy to gain a thorough knowledge of his subject; the deep personal interest in students that motivates him to do the best teaching of which he is capable; the scope of knowledge to contribute to both the professional and personal development of his students; a forceful personality embodying those traits which enable him to work gracefully with people; and great enthusiasm for his work.

The author is not content with mere generalities. He goes into the details of planning courses and individual lessons, discusses the various available instructional aids, from lecture notes and blackboards to projection lanterns, and gives pertinent advice on conducting class sessions. All these discussions are enlivened by effective diagrams and illustrations.

He deals understandingly and sympathetically with student learning activities and presents a very commonsense chapter on the ticklish subject of educational measurements, in which there are many helpful suggestions about tests and examinations.

> Teaching has been accorded professional status, and professional ethics dictate that the teacher be left largely to his own devices. Professional ethics, however, should not be used as a refuge for the incompetent, nor should they condone and protect poor practice. If it is to be considered unethical for the individual to be interfered with in his work, it should also be considered unethical for that individual to slight his work.

In view of this, teachers are urged to criticize their own efforts continuously. Even "experienced" teachers need this, since length of service is no criterion of teaching success.

The training of teachers is one of the most important aspects, and on this score the author seems to be just a trifle pessimistic. He outlines several ways in which to help the new instructor, but he confesses that in practice these are not always sufficient. Most of the burden must be on the instructor himself, and perhaps his best recourse will be to read this little book!

[1] Published by McGraw-Hill Book Co., Inc., New York. Price, $0.60.

EARLY WORK ON THE DISPLACEMENT OF METALS AND METAL OXIDES

VLADIMIR N. IPATIEFF
Universal Oil Products Company, Riverside, and Northwestern University, Evanston, Illinois

VLADIMIR N. IPATIEFF

WE REGRET to inform our readers of the death of Professor Vladimir N. Ipatieff, which occurred on November 29, 1952, following a short illness, just eight days after his 85th birthday.

Funeral services were held at Chicago, December 1, for this world-famed petroleum chemist and director of chemical research for Universal Oil Products Company. He had been associated with Universal for more than 23 years—ever since he left his native Russia with whose policies and philosophies he disagreed.

He was generally regarded as the world's foremost authority in the field of catalysis and it was in this area that he devoted the major portion of his remarkable career. He is particularly known for his fundamental research and development on the processes which contributed to the development of high octane aviation gasoline—alkylation, polymerization and isomerization.

It was the development of these processes which enabled the Allies to gain the upper hand in the aerial war against Germany during World War II and led to the eventual destruction of German aerial strength.

In addition to his work for Universal at their Riverside, Illinois, laboratories, Professor Ipatieff was an active member of the faculty of Northwestern University, where he set up and supervised the Ipatieff High Pressure and Catalytic Laboratories, the only unit of its kind on the campus of any American university.

Professor Ipatieff was born in Moscow in 1867, and attended a number of military schools there. But throughout his schooling period he maintained an active interest in chemistry and after his graduation from the Michaelovsky Artillery Academy continued with the academy as a chemistry professor. Four years later, in 1896, the academy sent him to Germany and France for further study in chemistry.

While he achieved world-wide recognition for his chemical research in Russia, it was in the latter period of his life, spent here in the United States, that he felt he reached his greatest accomplishments.

During his lifetime he received many honors and awards for his scientific accomplishments, the most recent of which were two French citations which he received last year—one making him a Chevalier of the Cross of Lorraine and a Companion of the Resistance, and the other an appointment as a member of the French Association of the Knights of Cyprus and Jerusalem. Both these awards were granted in recognition of his work on high octane aviation gasoline during the last war.

Professor Ipatieff's widow, Varvara, outlived him by only ten days. They are survived by a son and a daughter, both of whom live in Russia.

A RECENT issue of *Chemical and Engineering News* (*1*) contains an article on "The chemical ore reduction process." The article describes a new method which makes it possible to separate metals from solutions of their salts by means of hydrogen at higher temperatures and pressures. It appears that three plants will be built to utilize this particular method.

It is most gratifying to me to learn of this development since my co-workers and I have published some 30 articles on this particular subject over the period from 1909 to 1930. These have been published chiefly in *Berichte der Deutscher Chemischen Gesellschaft.* It is my hope that this particular work has served as the foundation for the technological developments which have just been announced.

My colleagues at Northwestern University and at Universal Oil Products Company have urged me to review the work we carried out from 1909 to 1930 with the idea that a better understanding of the reactions taking place would be derived from such a presentation.

Since the displacement of metals from aqueous solutions of their salts by hydrogen involves the utilization of high pressures, it is first of all interesting to recall the great struggle that I had in convincing chemists, as well as engineers, that the application of high pressures not only provides a new tool for effecting reactions heretofore believed impossible, but it also provides a safe and simple means for carrying them out.

DISPLACEMENT OF METALS

In 1908 I had just completed a series of successful experiments on the hydrogenation of organic compounds under pressure, and conceived the idea of applying this method to the reactions of inorganic compounds. Together with my assistant, V. N. Verkhovsky (*2*), we started a study of the reaction of hydrogen under pressure with solutions of various salts. Prior to this, this reaction was carried out in 1859 by Academician M. N. Beketoff (*3*), who succeeded in replacing silver and mercury from dilute solutions in the presence of hydrogen at 10 atm. pressure; however, he was unable to precipitate copper or lead under these conditions. He indicated that copper, as well as some of the other metals, could be displaced from their solutions in the presence of platinum black; however, in this case one can easily suspect a catalytic reaction.

As can be seen from the table of the electromotive force of the metals (Table 1) each member displaces

TABLE 1
Electromotive Series of the Metals

Zn 0.8	Sn 0.14	As −0.3
Fe 0.44	Pb 0.13	Cu −0.34
Cd 0.4	H_2 0.0	Ag −0.8
Co 0.33	Sb −0.1	Pt −0.8
Ni 0.25	Bi −0.2	Au −1.68

from a salt solution each succeeding member, that is, metallic zinc replaces hydrogen, copper replaces silver, and platinum replaces gold, etc. It should be noted, however, that this reaction is not reversible and that the metals above hydrogen are soluble in acids while the metals below hydrogen are insoluble in acids.

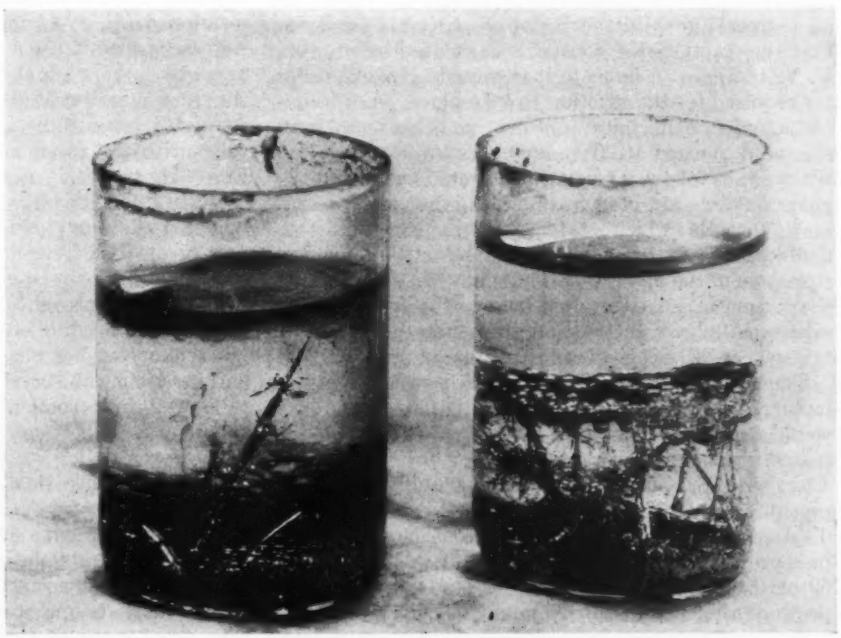
In 1892 Tammann and Nernst(*4*) tried to precipitate copper from copper sulfate by using hydrogen under pressure up to about 40 atm. In a great many cases their experiments terminated in an explosion, while those that survived the experimental conditions still did not show separation of copper from copper sulfate. Until our work in 1908 this was the last attempt to separate copper and other metals by means of hydrogen.

In our experiments we started with the study of the effect of hydrogen pressure on the displacement of metals from their salts at room temperature. A 0.1 *N* solution of copper sulfate was placed in a glass or quartz tube and inserted into the Ipatieff autoclave, and then hydrogen was pumped in. In order to prevent the loss of water by evaporation the tube was equipped with a stopper having a capillary opening. In these experiments it was found that although pressures of 200, 580, and 600 atm. were used, and the experiments were carried out for several days, there was no trace of any separation of copper from the solution. In other experiments we have tried other salts of copper, as well as the salts of other metals, such as nickel, cobalt, and cadmium. However, despite the use of high pressure, none of these experiments showed any displacement of the metals from their solutions at 25°C. We decided to heat the bomb, or autoclave, to higher temperatures, and only then did we observe deposits at the bottom of the tubes. The composition of these deposits depended upon the temperature at which the experiment was carried out. For example, copper sulfate at 90°C. and 25 atm. pressure gave a crystalline material having a composition $CuSO_4 \cdot 2Cu(OH)_2$, while after longer times of contact the separation of Cu_2O was found to take place. When the experiment was continued for 40 to 50 hours crystals of copper separated out along with Cu_2O, while after seven days of heating the only product found was metallic copper, according to the equation:

$$CuSO_4 + H_2 \rightarrow Cu + H_2SO_4$$

At 200 atm. pressure and 90°C. pure copper separates out readily. The effect of temperature is quite pronounced. For example, at 50°C. and 700 atm. pressure, copper sulfate does not produce cuprous oxide; however, at 60°C. and 600 atm., cuprous oxide does precipitate out. These experiments showed that the formation of the basic salts and metal oxides takes place through the hydrolysis of the salts, while the precipitation of the metals by means of hydrogen is a separate reaction. This was proved by the addition of sulfuric acid to a solution of copper sulfate, thereby providing sufficient hydrogen ions to repress hydrolysis and to result in the precipitation of pure copper by treatment with hydrogen. It was finally found that for each metal there is a certain critical temperature and pressure which permits the displacement of the metal from its solution by means of hydrogen.

Further experiments showed also that at atmospheric pressure an increase in temperature does not bring out the separation of the metal; thus the cooperative action of both temperature and pressure is essential. It should be noted, of course, that when we talk of critical temperatures and pressures, there is no connection at all with the critical temperatures and pressures governing the behavior of gases. As stated above, the hydrolysis reaction requires one set of conditions, and this is



Copper Crystals Recovered from Autoclave

V. N. Ipatieff in 1918 as a Lieutenant General of the Russian Army

followed by the precipitation of the metal from the solution. Our definition of the critical temperature for the replacement of a given metal from a water solution of its salt is the temperature at which a metal is precipitated out rapidly and to completion at a minimum hydrogen pressure. If one makes the supposition that the reaction of the replacement of metals takes place at all temperatures, but very slowly, it should be noted that the reaction-rate constants calculated by my son, V. V. Ipatieff (*5*), indicate that exceedingly long times are required for the reaction to take place. For example, in order to precipitate one per cent of the metal from a normal solution at 20°C. and 100 atm., the required times are as follows: bismuth, 37 years; antimony, 160 years; and arsenic, 1140 years. Using the very approximate formula which states that the reaction rate doubles for each 10°C., we should expect that the precipitation of the metal would take place in a reasonably short time at relatively low temperatures. However, experiments have shown that the separation does not take place until a certain critical temperature is reached.

The same reasoning can be applied to the pressure factor; if one considers electropotential values one would expect that bismuth, for example, could be displaced from a solution at relatively low pressures and at a temperature of 175°C. However, actually there is no precipitation at 15 atm. of hydrogen pressure, and the displacement only takes place at considerably higher pressures. In an actual experiment it was found that 60 per cent of the bismuth is precipitated at 150 atm. pressure after heating for 23 hours at 175°C. It is interesting to note that further increases of pressure raise the separation extent to only 62 per cent and complete precipitation of bismuth from a solution requires considerably more severe conditions.

As is known, in order to explain the reaction of the displacement of metals by hydrogen Nernst proposed an equation which makes it possible to calculate the pressure required for carrying out the reaction. This pressure must be greater than the electrolytic solution pressure of the metal.

$$\log P = \frac{\pi \times Ne}{0.058} \log p$$

Where P is the electrolytic solution pressure, π is the electropotential difference, Ne is the potential of the cation, and p is the osmotic pressure of the cation. However, this equation does not include the effect of temperature and therefore requires some modification. I recall a lecture which I delivered before the German Chemical Society in 1923. Following the lecture, Dr. Nernst invited me to come to his laboratory in order to discuss further the mechanism of the reaction, but after long discussions we could not arrive at a reasonable hypothesis. I also recall that at the Seventh International Chemical Congress in London in 1909, Arrhenius, who was the Chairman of the meeting, indicated that some changes must be made in our theoretical viewpoints on the calculation of the electrolytic solution pressure of metals. For example, for the separation of cadmium from its nitrate, the calculated required pressure of hydrogen is 10,000 atm.; however, our experiments showed that a 2-N solution of this salt at 270°C. and a pressure of 220 atm. in a quartz tube yields hexagonal crystals of metallic cadmium in 10 hours.

It should be noted that in our experiments the choice of required temperature also governed our choice of the reaction vessel. At temperatures in excess of 250°C. we were compelled to use a quartz tube instead of an ordinary glass tube, while at temperatures in excess of 300°C. the reaction was carried out in gold and platinum tubes. At high temperatures water vapors act on the quartz tube, with the result that under these conditions we were able to obtain hexagonal quartz crystals. Very recently I had the pleasure of seeing the beautiful large quartz crystals prepared by the excellent method worked out by the Bell Telephone Laboratories for the preparation of large-size quartz crystals at high temperatures and high pressures (A. C. Walker and E. Buehler (*6*)). The procedure involves the seeding of an aqueous phase above a silica phase with a minute quartz crystal and permitting the crystal to grow evenly and slowly.

Table 2 shows some of the typical results obtained in the precipitation of copper and other metals by means of hydrogen under pressure. The indicated temperatures and pressures do not represent the critical temperatures and pressures.

These experiments showed that the addition of an acid to a neutral solution of the salt has a profound effect on the displacement of the metal by hydrogen at at constant temperature. For example, a neutral solution of cupric acetate at 100°C. and 40 atm. hydrogen pressure yielded cupric oxide; when the tempera-

TABLE 2
Displacement of Metals from 1/10 *N* Aqueous Solutions

Salt	*Temp., °C.*	*Pressure, atm.*	*Product analysis*
$CuSO_4$	20	580	No Cu
$CuSO_4$	160	118	100.0% Cu
$CuSO_4$ + H_2SO_4	122	120	99.93% Cu
$NiSO_4$	207	123	99.74% Ni
$CoSO_4$	230	150	100.0% Co
$Pb(C_2H_3O_2)_2$	240	150	100.0% Pb
$Bi(NO_3)_2$	248	150	100.0% Bi
$K_2H_2Sb_2O_7$	330	200	100.0% Sb

ture was increased to 180–190°C. at the same pressure only traces of cuprous oxide were found in cupric oxide. In order to obtain the separation of metal with copper it is essential that the solution contain a definite concentration of hydrogen ions. As long as the concentration of the acid does not exceed 75–76 per cent (12.5-*N*), hydrogen displaces only cuprous oxide. Higher concentrations of acid in the salt solution stop the hydrolysis and result in the separation of pure metallic copper. In this particular case the solution at 20°C. has a pH of 1.12, which corresponds to a hydrogen ion concentration of 0.075.

An investigation (*7*) of the solution remaining after the precipitation of metal from copper sulfate showed that it also contained some cuprous sulfate, Cu_2SO_4. The question was immediately raised as to what conditions are required for the conversion of cupric sulfate into cuprous sulfate. A number of experiments showed that under no conditions was it possible to obtain only Cu_2SO_4; moreover, it was not possible to separate metallic copper by hydrogen without leaving in the solution some cuprous and cupric ions, although in some cases the amounts present were negligible. This would indicate that under the experimental conditions the displacement of copper by hydrogen never goes to completion, and the residual solution still contains traces of copper ions. Table 3 gives the data which confirm the precipitation of metallic copper from cupric sulfate with a formation of both cuprous and cupric ions in the residual solution. The reactions which govern this are most likely the following:

$$2\,CuSO_4 + H_2 \rightarrow Cu_2SO_4 + H_2SO_4$$
$$Cu_2SO_4 + H_2 \rightarrow 2\,Cu + H_2SO_4$$
$$CuSO_4 + Cu \rightarrow Cu_2SO_4$$

The last reaction can be written:

$$Cu^{++} + Cu \rightarrow 2\,Cu^{+}$$

There is no doubt that the entire system appears to be in a dynamic equilibrium involving hydrogen and both cuprous and cupric ions.

The above experiments were carried out in the Ipatieff rotating autoclave or in autoclaves equipped with mechanical agitators. For example, a solenoid in the head of the bomb was connected to a rod provided with horizontal discs; the rod was moved continuously up and down through the solution by action of the solenoid. Other autoclaves, equipped with stirrers revolving at 200 r. p. m., also provided for a reasonably good mixing of the solutions.

TABLE 3
Effect of Hydrogen Ion Concentration on Separation of Copper from 1.0 *N* Copper Sulfate Solutions. Time: 24 hours, Temp.: 100°C.

H_2 Pressure, atm.	*Conc. of H_2SO_4*	*Distribution of Cu based on original $CuSO_4$*		
		as Cu^{++}	*as Cu^{+}*	*as Cu metal*
100	None added	54	15	31
100	1.0 *N*	30	35	35
100	2.0 *N*	52.5	27	20
100	4.0 *N*	55.5	22	22.5
100	14.0 *N*	40.5	47	12.5
20	None added	37.5	40	22.5
20	1.0 *N*	33	53.5	13.5
20	2.0 *N*	41	56	3
20	4.0 *N*	77.5	22.5	0
20	14.0 *N*	98.5	1.5	0

SEPARATION OF METALS

In view of the fact that various metals have different electrode potentials, and since it was indicated that different temperatures and pressures are required for displacing the metals from their solutions, it would be reasonable to expect that the action of hydrogen under pressure on solutions containing different metal ions could result in the separation of the metals. It would be expected that the metal with the lowest electrode potential would be separated first. For example (*8*), if one has a mixture of copper sulfate and zinc sulfate solutions, or copper sulfate with iron salts, such as Mohr's salt [$(NH_4)_2SO_4{\cdot}FeSO_4{\cdot}6H_2O$], the initial precipitate is metallic copper. The separation of copper occurs at temperatures and pressures somewhat higher than those required for precipitation of copper from solutions containing no other metal ion. In order to prevent the hydrolysis of other salts it is necessary in some cases to add sulfuric acid to the system.

A number of experiments have been carried out by my son, V. V. Ipatieff (*9*), on the separation of arsenic, bismuth, and antimony at 100 atm. hydrogen pressure and temperatures ranging from 175 to 225°C. In order to separate arsenic from bismuth, a 2-*N* hydrochloric acid solution was employed; at the end of 18 hours arsenic had separated completely, while the residual solution contained the bismuth chloride.

In the separation of arsenic from antimony he used an apparatus equipped with a stirrer and a solution of the chlorides of the metals with a normality of 0.5 and an additional hydrochloric acid concentration of 2 *N*. At the end of 22 hours of heating, all the arsenic was recovered in the deposit, while the antimony was retained in the solution. It was found, however, that if the system was subjected to further heating after the precipitation of arsenic there was some deposit of antimony; however, there was no re-solution of the arsenic deposited in the early stages of the experiment. The separation of antimony from bismuth takes place only partially, an observation which may be explained by the very small difference in electropotential between the two metals in solutions of equal concentration. Fur-

ther experiments made by V. V. Ipatieff, concerned the separation of the metals of the platinum family.

BASIC SALTS AND METAL OXIDES

The precipitation of crystalline basic salts and metal oxides under hydrogen pressure occurs via hydrolysis. In this case one may also ascribe to water an oxidizing action which becomes more and more intense as the temperature is increased.

In our experiments we approached 360°C. as the upper limit in order to maintain a liquid phase. We were unable to precipitate metallic iron because its critical temperature of precipitation is above 360°C., and under those conditions water tends to oxidize the iron back to an oxide. The same applies to other members of the iron group.

Iron. A solution of iron nitrate produced octahedral crystals of Fe_3O_4; the same well-developed crystals were obtained from potassium ferrocyanide. However, in the latter case we also obtained in the solution some formic acid, ammonia, and carbonic acid. The formation of these products is to be expected.

Of considerable interest was the formation of crystalline basic salts of iron, produced from ferric phosphate (*10*). The products contained both di- and trivalent iron in the molecule, having the general formula of

$$Fe_n^{+++}\,Fe_m^{++}\,(PO_4)_x \cdot yH_2O$$

and, depending upon the composition, the resulting salts had various colors ranging from green to blue to black. These products correspond to the naturally occurring mineral vivianite. Ferric arsenate at 355°C. and 260 atm. produces scorodite ($FeAsO_4 \cdot H_2O$).

Manganese (*11*). Both potassium permanganate, $KMnO_4$, and manganese nitrate, $Mn(NO_3)_2$, at 300°C. and 150 atm. yielded beautifully developed cubic crystals of Mn_3O_4, identical with the natural mineral hausmannite. Manganese chloride at 360°C. and 350 atm. gave $MnO \cdot H_2O$, which is readily oxidized by air.

Chromium. Chromium nitrate gave the crystalline chromium oxide hydrate $Cr_2O_3 \cdot H_2O$.

Nickel and Cobalt (*12*). An aqueous solution of nickel cyanide, $Ni(CN)_2$, at 150 to 160°C. and a hydrogen pressure of 80 atm. produces a crystalline deposit of the anhydrous NiO; this compound has yet to be obtained from other salts of nickel. Furthermore, this experiment shows that we are as yet unable to obtain a higher nickel oxide, Ni_2O_3, contrary to our experience with cobalt. In the latter case, a solution of the cobalt salt in the presence of hydrogen under pressure and 260°C. produces Co_2O_3.

Copper (*13*). It was found that 1 and 2-*N* solutions of copper nitrate at 168°C. and an initial hydrogen pressure of 100 atm. gave a basic salt insoluble in acids and mixtures of acids. The salt was formed in well-developed green crystals analyzing as $Cu(NO_3)_2 \cdot 3Cu(OH)_2$, the structure of which, according to Werner's theory, is:

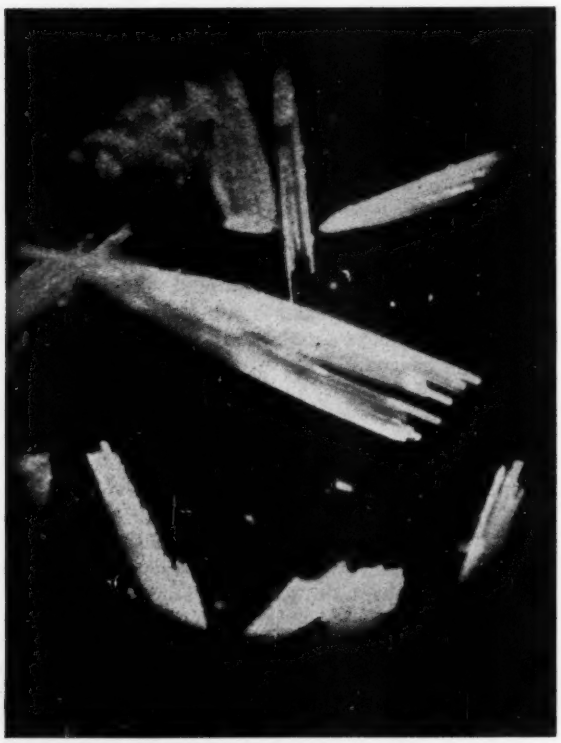
$$Cu\begin{pmatrix} OH & Cu \\ OH & \end{pmatrix}_3 \cdot (NO_3)_2$$

This product is found in nature under the name of gerhardtite. It should be noted that if one substitutes nitrogen for hydrogen, the reaction does not take place.

Experiments with copper arsenite under the same conditions gave the mineral domeykite, Cu_3As.

Zinc (*14*). According to the electrolytic solution pressure equation, the replacement of zinc from the water solutions of its salts requires a hydrogen pressure in excess of 50,000 atm. Our experiments have shown that a 1-*N* solution of zinc nitrate at 350°C. and 350 atm. hydrogen pressure produces the precipitation of metallic zinc, which deposits upon previously formed crystals of zinc oxide. At lower temperatures zinc nitrate produces well-developed needles of zinc oxide with a slight greenish coloration. Zinc sulfate at 290°C. and 250 atm. yielded zinc sulfide crystals, ZnS, identical with the naturally occurring mineral zinc blende. It is interesting to note that in several experiments on the precipitation of zinc oxide in quartz tubes, well-developed crystals are formed which upon analysis showed a composition of $ZnSiO_3 \cdot 3H_2O$. This compound can be obtained also from a 2-*N* solution of zinc nitrate if it is heated to 320°C. at 250–300 atm. using unpolished quartz tubes. Similarly it was found that upon heating magnesium and manganese salts with water glass similar crystalline silicates are formed.

The results discussed above provide an indication of the tremendous scope of the study of reactions of inorganic compounds at high pressures and temperatures.



Zinc Silicate Crystals Formed by the Reaction of Zinc Nitrate with Quartz (Ber., 59, 1418 (1926))

Such a study should explain not only the mechanism of these particular reactions but also the formation of naturally occurring minerals. Such investigations would therefore be of considerable help in geological work. As a matter of fact, in 1912, following one of the reports of the work described above, a well-known geologist predicted that such work would have in the future not only experimental but also commercial application. This was a correct prediction; just at the time of my departure for the United States in 1930 I was asked by the Bayerische Stickstoff Werke in Germany to undertake a continuation of the experiments on the displacement of metals from solutions, since even at that time there was considerable interest in the production of copper from copper sulfate solutions.

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A CONVENIENT APPARATUS FOR THE DISTILLATION OF SOLIDS[1]

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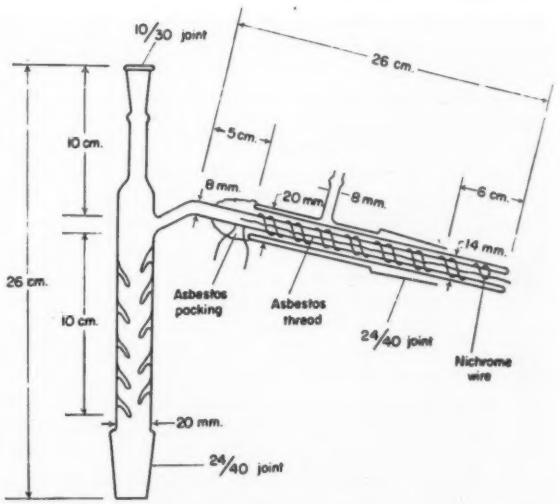
WHEN attempts are made to distill a solid or a mixture of solids which melts at 45–50°C. or higher and crystallizes easily on distillation, difficulties are often encountered in preventing the material(s) from solidifying in and clogging the sidearm of the standard distillation equipment.

Compound	*Melting Point, °C.*	*Boiling Point, °C./2 mm.*
Benzophenone	48.2– 48.8	124–126
Phenyl benzyl ketone	59.6– 60.5	131–132
Triphenylmethane	93.4	169–172
Benzoic acid	121.2–121.8	125–128
Tetraphenylethylene	223.6–224.2	180–185

To overcome these difficulties, the apparatus shown in the accompanying diagram was designed. It may be seen that the apparatus consists of a modified Vigreux column with the sidearm electrically heated and equipped with a vacuum take off. In wrapping the sidearm, four feet of Nichrome alloy wire, B & S No. 20, 0.672 ohms/foot, were used. In use, the lead wires from the sidearm are connected to a Variac and it has been found that a setting of ten volts on the Variac produces enough heat to keep the solids fluid during distillation. Employing this piece of equipment, the compounds listed in the table were conveniently and easily distilled.



[1] This work was done under Contract No. AT(30-1)-670 between the Atomic Energy Commission and the University of Pittsburgh.

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ELECTROLYTIC CAUSTIC AND CHLORINE INDUSTRIES[1]

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CHLORINE ranks fifth among big-tonnage chemicals. Toward the end of 1951 daily capacity in the United States was just short of 7000 tons. A 50 per cent increase in capacity is under way. In *Chemical Engineering* last month (**59,** 175 (1952)) 36 new chlorine plants were listed. An estimate of the capacities of these new plants and the processes to be used compares with existing plants as follows:

	Existing Plants	*New Plants*
Diaphragm cells	84 per cent	53.5 per cent
Mercury cells	7	37.2
Downs sodium cells	8	7.5
The nitrosyl chloride	1	0.8
A new modified Deacon process	...	1.0

Note the increased popularity of mercury cells. These cells produce directly a concentrated caustic soda of the highest purity, which is the first choice of the rayon industry and is easier to sell than any other grade.

Three Niagara Falls cell designs are contributing to the present expansion. The Downs cell produces metallic sodium and chlorine from fused sodium chloride. The Hooker diaphragm cell and the Mathieson mercury cell both make chlorine and caustic soda. This paper covers only electrolytic caustic and chlorine, so the sodium cell will not be discussed.

In a diaphragm cell an asbestos diaphragm separates a graphite anode from a steel cathode. Brine is fed into the anode compartment and flows through the diaphragm. A direct current deposits chlorine gas at the anode, hydrogen gas and sodium hydroxide around the cathode. Hydroxyl ions tend to migrate toward the anode, but the flow of brine through the diaphragm counteracts this tendency. The liquor leaving the cell is approximately 11 per cent NaOH and 15 per cent NaCl. It is evaporated until the NaOH content is 50 per cent by weight. Salt crystallizes out, is filtered, redissolved, and recycled. Standard 50-per cent diaphragm-cell caustic contains 1.1 per cent salt and other impurities. A number of purification methods are in use for improving the quality of this NaOH. The raw material is rock salt dissolved in water or brine from wells. In either case the brine must be treated to remove $CaSO_4$ and magnesium. The theoretical voltage of the diaphragm cell is 2.3 volts and commercial cells operate at around 3.5 volts.

In a mercury cell, direct current is passed through brine from a graphite anode to a mercury cathode, forming chlorine and sodium amalgam. This reaction requires 3.1 volts theoretically and commercial cells operate at 4.0 to 4.5 volts. The sodium amalgam flows to another compartment where it reacts with water to form caustic soda and hydrogen gas. The mercury is recycled to the first compartment. Solid salt is required for this process to maintain a circulating brine system close to saturation. Brine treatment is somewhat more involved than with the diaphragm-cell process. New mercury-cell plants will use designs of deNora, Mathieson, Solvay, or I. G. Farben. The I. G. Farben design was published in FIAT reports (U. S. Office of Technical Services, PB33221 "FIAT Report No. 816," Horizontal Mercury Chlorine Cell). The deNora cell was described in recent transactions of the Electrochemical Society (*Trans. Electrochem. Soc.*, **97,** 346 (1950)). No details have been disclosed on the Solvay cell. The latest model of the Mathieson cell will be described later.

The Dow Chemical Company is making the largest expansion with diaphragm cells of their own design. So far, they have not made their cell design available to the public.

HOOKER CELL

The diaphragm cell designed by the Hooker Electrochemical Company probably makes more chlorine than any other cell. Most of the expansion during World War II and since has been with Hooker cells. Their development started in 1903 when the Development and Funding Company, predecessor of Hooker Electrochemical Company, was formed to develop the caustic-chlorine cell patented by Clinton P. Townsend. During the years many improvements were made. Two major inventions have been the deposited-diaphragm, developed 1925–29, and the basic Type S cell designed in 1929–30. Now the Type S cell operates at 10,000 amperes, the Type S-3 at 20,000 amperes and the Type S-3A up to 30,000 amperes. The Type S cells are recommended for plants producing up to 75 tons of chlorine per day. However, for larger plants the larger cells are more economical.

A diagram of the Hooker Type S cell is shown in Figure 1. The cell has three main parts. The anodes, which are flat blades of impregnated graphite, have their lower ends embedded in a lead slab cast in the concrete bottom, *C*. The middle cathode section, *B*, is a rectangular steel frame with rows of parallel fingers

[1] Presented at the 121st Meeting of the American Chemical Society, Buffalo, New York, April, 1952.

projecting inwardly from two opposite sides and spaced to alternate with the anodes. The cathode fingers are made of wire screen. The diaphragm is deposited on the cathode by immersion in a suspension of asbestos fibers in a liquid of suitable specific gravity and application of vacuum to the hydrogen outlet. When a certain vacuum bas built up, the cathode is lifted out of the depositing tank and air is drawn through the diaphragm to compact and dry it. The cathode section rests on the cell bottom. A concrete top, *A*, completes the assembly. The brine inlet and chlorine gas outlet are in the top section. Hydrogen gas and cell liquor leave from suitable connections on the cathode section.

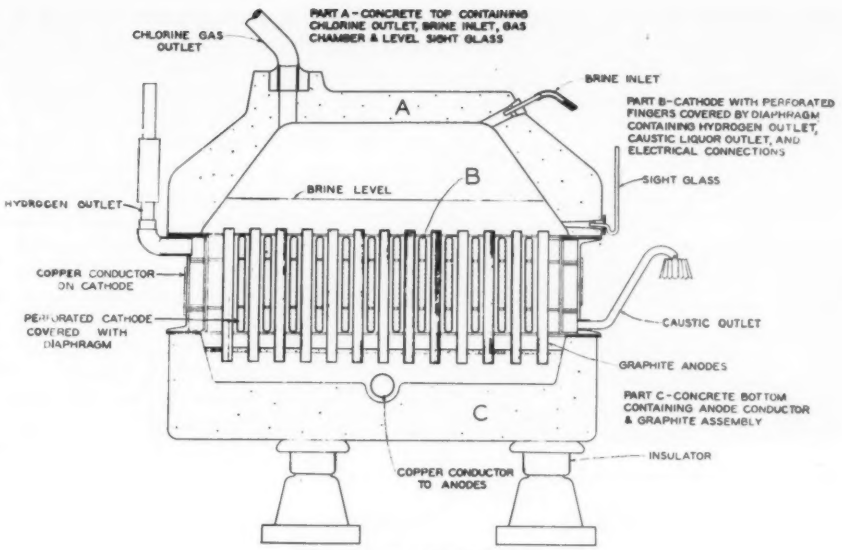
CHLORINE GAS OUTLET
PART A - CONCRETE TOP CONTAINING CHLORINE OUTLET, BRINE INLET, GAS CHAMBER & LEVEL SIGHT GLASS
BRINE INLET
PART B - CATHODE WITH PERFORATED FINGERS COVERED BY DIAPHRAGM CONTAINING HYDROGEN OUTLET, CAUSTIC LIQUOR OUTLET, AND ELECTRICAL CONNECTIONS
A
B
BRINE LEVEL
SIGHT GLASS
HYDROGEN OUTLET
COPPER CONDUCTOR ON CATHODE
PERFORATED CATHODE COVERED WITH DIAPHRAGM
CAUSTIC OUTLET
GRAPHITE ANODES
PART C - CONCRETE BOTTOM CONTAINING ANODE CONDUCTOR & GRAPHITE ASSEMBLY
C
INSULATOR
COPPER CONDUCTOR TO ANODES

Figure 1. Hooker Type S Cell

As in all diaphragm cells, the anodes gradually wear out and the diaphragms gradually plug and require renewal. Three diaphragms are used during the anode life.

It is one of the important design features of the cell that the brine feed may be held constant for a given current throughout the life of the cell, and that the anolyte brine level within the cell adjusts itself according to the porosity of the diaphragm to maintain a constant rate of flow through the diaphragm. Thus, the sodium hydroxide concentration is constant, except for a minor variation due to the change of evaporation rate as the cell temperature changes. Concentrations of 120 to 160 g./l. NaOH may be maintained to meet the special requirements of local conditions. Current efficiency and graphite life tend to increase as the sodium hydroxide concentration is reduced; however, steam requirements for evaporation also tend to increase. Hence the economical operating range depends on local power and steam costs. Generally, 130 to 140 g./l. NaOH are most economical for conditions in this country.

An installation of Type S-3A cells is shown in Figure 2. The Hooker Type S-3 and Type S-3A cells have the same fundamental arrangement as the Type S cell except that the anode and cathode area are doubled. The copper is heavier on the Type S-3A and the cell is turned 90°. The S-3 is rated 20,000 and the S-3A operates satisfactorily at 30,000 amperes. The cells are arranged back to back in double rows with an intermediate structure carrying the brine, chlorine, and hydrogen mains. The cell liquor collecting line runs along the floor in front of the cells.

The Type S cell operating at 10,000 amperes has an average voltage of 3.72 volts and 96 per cent current efficiency. The Type S-3 and S-3A cells operating at 20,000 amperes have an average voltage of 3.65 volts and 96 per cent current efficiency.

It is interesting to note the advantage of large cells on the saving of floor space. The 10,000-ampere cell produces 9.2 lb. of Cl_2 per day per square foot, including aisles, while the 20,000 ampere cell produces 15.1 lb.

Figure 2. Hooker S-3A Cells

MERCURY CELL

Demonstration. The mercury cell process can be demonstrated with the relatively simple equipment shown in Figure 3.

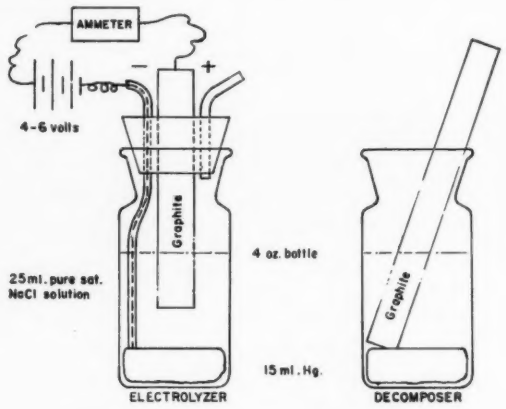

Figure 3. Demonstration of Mercury-Type Cell

Electrolyze 10 min. at 10 amp. Separate 0.136 per cent Na amalgam (theor.) from brine and cover with dilute NaOH solution. Note absence of H_2 bubbles until graphite touches amalgam surface.

(1) Place 15 ml. of clean mercury in a 4-oz. wide-mouth bottle and add approximately 25 ml. of pure, filtered NaCl solution.

(2) Suspend a graphite or platinum anode in the brine. Make a cathode connection with an insulated wire dipping into the mercury. Pass 2 amperes from a storage battery through the brine for approximately 10 minutes and the usual concentration of 0.1 to 0.13 per cent Na amalgam will be formed. This takes place in that part of a mercury cell called the electrolyzer.

(3) Now stop electrolysis and separate the amalgam from the brine into another bottle containing water, or preferably dilute sodium hydroxide solution. Notice that there is little or no reaction between the sodium amalgam and water. Now lower a piece of graphite through the water. A brisk evolution of H_2 will occur when the graphite touches the amalgam. This shows the reaction in what is called the decomposer. It is actually a short-circuited electrolytic cell, sodium amalgam being the anode and graphite the cathode.

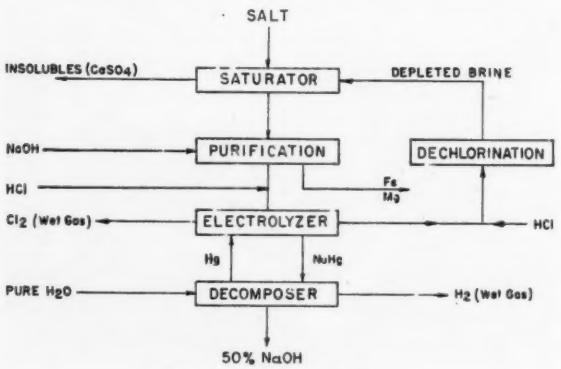

Figure 4. Mercury Cell Process

A simplified flow sheet of the mercury cell process is shown in Figure 4. Note that there is a mercury cycle and a brine cycle. Consider the brine cycle starting with pure brine entering the cell. It enters the electrolyzer at 310 g./l. NaCl and overflows at 270 g./l. NaCl. Pure brine is essential to the successful operation of the mercury cell. Brine leaves the electrolyzer saturated with chlorine. This is removed by adding hydrochloric acid and pumping the brine through a vacuum chamber where the chlorine flashes off. The dechlorinated brine is resaturated with solid salt, then made alkaline with caustic soda to precipitate impurities such as Fe and Mg. Mathieson engineers have found that the cell will tolerate brine saturated with $CaSO_4$ if it is acidified. The resaturated brine is settled and filtered. The pH is then adjusted to 2.5–3.0 and returned to the cells.

The reason for taking only 10 to 15 per cent of the NaCl content out of the brine before resaturation and the reason for purification involve some fundamental electrochemistry at the amalgam surface in the electrolyzer.

H^+ ions and Na^+ ions are both tending to deposit at the mercury cathode. The overvoltage of hydrogen is high, whereas the sodium potential is lowered at a dilute amalgam surface. At a mercury surface conditions with a pure brine favor sodium deposition. However, conditions arise which lower hydrogen overvoltage at the mercury surface and H_2 is evolved. This contaminates the chlorine. Above 5 per cent of H_2 in Cl_2 may explode, so this condition must be avoided. Brine impurities such as Fe, Mg, Cr, V, etc., are particularly harmful. In normal operation the cell chlorine should contain less than 1 per cent of H_2.

The concentration of the caustic soda leaving the decomposer is controlled by the flow of water to it. To make pure caustic, pure water must be used. Caustic soda of 50 per cent concentration or better leaves the decomposer. Amalgam enters the decomposer at 0.1 to 0.2 per cent Na and should leave at less than 0.01 per cent Na.

The Mathieson Cell. In 1895 The Mathieson Alkali Works, Inc., installed a circuit of 54 Castner rocking cells at Saltville, Virginia. On Thanksgiving Day, 1897, Mathieson started Castner rocking cells at Niagara Falls. These cells have undergone many mechanical improvements but are still operating.

In 1935 the development of stationary-type mercury cells was started. All of the four basic modifications of cell design have been tested: (*a*) an electrolyzer with a continuous steel bottom, (*b*) an electrolyzer with a protected bottom, (*c*) a horizontal decomposer, parallel to the electrolyzer, and (*d*) a vertical decomposer in the form of a tower. Mathieson engineers concluded that the continuous steel electrolyzer bottom and the vertical decomposer are most economical for American conditions.

Other noteworthy features of the Mathieson cell include: (*a*) a one piece cover for the electrolyzer, (*b*) a metallic lead-in to the anode blocks, (*c*) anodes that are adjustable during operation, and (*d*) current densities over 3 amperes per square inch based on the mercury cathode surface.

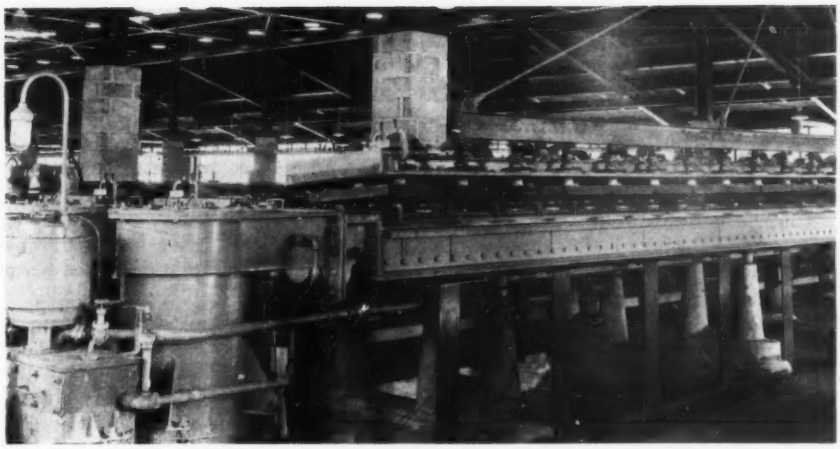

Figure 5. Mathieson E-8 Cell with Electrolyzer Cover Raised. Mercury Pump and Decomposer at Left

The latest Type E-8 Mathieson cell is shown in Figures 5 and 6. The electrolyzer bottom is a steel channel made from a 24-inch I-beam. The sides are 8-inch channels, hard-rubber-covered on the upper flange and web. The ends are closed by the inlet and outlet boxes, which are hard-rubber-lined cast iron.

The electrolyzer cover is a single piece of reinforced steel, rubber-covered on all surfaces exposed to chlorine. It rests on a soft rubber gasket and C-clamps are used to make a gas-tight seal. It is handled by a crane. The anodes are supported from the cover. Lead-in posts pass through the cover and are sealed with flexible rubber seals. By turning a supporting nut the distance between the anode and cathode can be readily adjusted. This is a weekly operating procedure.

The anode construction has received a great deal of attention. A metallic lead-in has been developed. It is protected with a porcelain sleeve. There are two lead-ins for each anode block. Many configurations for the bottom of the graphite surface have been tried. Saw cuts are considered worth while. Anodes last about a year but are of various ages in each cell. The combination of bare-steel bottom, metallic lead-ins, adjustable anodes, and staggering of anode replacements has resulted in a very low slope to the volt-ampere curve. The current is limited to keep outlet brine temperatures below 85°C. Above this temperature there would be excessive attack on the rubber lining.

Figure 6. Mathieson E-8 Cells at Saltville Plant

The decomposer is fabricated from carbon steel. It is packed with lumps of crushed graphite. The packing is held between screens compressed with jack screws. A distributor plate spreads the amalgam evenly over the top of the packing. Deionized water is introduced at the bottom of the decomposer. Hydrogen and caustic outlets are located at the top of the decomposer. The mercury pump is a sump-type, centrifugal pump setting in a sump tank. The pump returns denuded amalgam to the inlet end of the electrolyzer.

The E-8 is nominally rated at 23,000 amperes, but has been tested at 27,500 amperes in the Niagara Falls pilot plant. It operates at 30,000 amperes at Saltville, Virginia, and McIntosh, Alabama.

CONCLUSION

The electrolytic alkali-chlorine industry is undergoing a 50 per cent expansion in 36 new plants. More than half of the new production will be with diaphragm cells. Dow cells and Hooker cells will be the most popular diaphragm cells. The latest Hooker cell has been described.

Mercury cells have increased in popularity because concentrated caustic soda of rayon quality is produced directly at the cells.

Solvay, deNora, I. G. Farben, and Mathieson cells are being installed. The latest Mathieson E-8 design has been described here for the first time.

ACKNOWLEDGMENT

The author wishes to thank the Hooker Electrochemical Company and the Mathieson Chemical Corporation for supplying information disclosed in this paper.

THE BASIC WORK OF FISCHER AND VAN'T HOFF IN CARBOHYDRATE CHEMISTRY[1]

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In the course of the year 1852—one hundred years ago—two boys were born in the region of the Rhine river valley whose contributions to the science of chemistry in their mature years were so important, so extensive and so fundamental that the Sugar Division of the American Chemical Society has designated its present meeting to be a memorial to them in this centennial year of their births. We desire to give expression in this public way to our unbounded admiration and appreciation of the services to mankind of Emil Fischer and J. H. Van't Hoff. We regard it as fitting that this memorial should be sponsored by the Sugar Division, because that very extensive field of organized modern science that is known today as carbohydrate chemistry is based upon ideas and discoveries that came from these two men.

J. H. Van't Hoff was born in Rotterdam August 30, 1852. Emil Fischer was born in Euskirchen, not far from Cologne, October 9, 1852. The parents of each of them were able to give the sons the very best educational opportunities. Van't Hoff received the Doctor's degree from the University of Utrecht in 1874, at the age of 22. Fischer received his Doctor's degree from the University of Strassburg in the same year; at the university he studied directly under Adolf Baeyer. Fischer discovered phenylhydrazine while in Strassburg in 1875, and in the same year he followed Baeyer, as assistant, to the University of Munich. He became professor at the University of Erlangen in 1881 and while there he discovered the first phenylosazones of the sugar group, in 1884. He received a call to the professorship in the University of Würzburg in 1885 and there he expanded his carbohydrate researches most energetically. It was from Würzburg in 1891, seven years after he had discovered the sugar phenylosazones, that he published his famous proof of the configurations of glucose and several of its relatives, including mannose, fructose, gulose, xylose, arabinose, saccharic acid, and glucuronic acid. This proof is the first published record of the establishment of a contact between the thoughts of Fischer and Van't Hoff. Both of them had attended

[1] Presented at the 122nd Meeting of the American Chemical Society, Atlantic City, September, 1952, at a meeting of the Sugar Division held in memorial celebration of the centennial year of the births of Emil Fischer and J. H. Van't Hoff.

[2] Dr. Hudson died on December 27, 1952.

Kekulé's lectures at the University of Bonn in the course of their journeyings as students but it is not known whether they met each other in those formative days. Van't Hoff, and independently J.-A. Le Bel, announced the theory of the asymmetric carbon atom in 1874, 17 years before it was used by Fischer in 1891; it is obvious today that, beginning with the discovery of glucose phenylosazone in 1884, Fischer's experimental researches in the sugar group were leading him inexorably to his fruitful use of the Van't Hoff-Le Bel theory seven years later. The amount of accurate experimental work that Fischer accomplished in this difficult virgin field during those seven years, first in Erlangen and later in Würzburg, is an early illustration of the ability, energy, and sound judgment that characterized his entire scientific career.

Beginning with Fischer's use of the Van't Hoff-Le Bel theory of the asymmetric carbon atom in 1891, the theory soon received such numerous further verifications, especially in the carbohydrate field, that it soon became generally recognized as the guiding mathematical (*i. e.*, geometric) principle of carbohydrate chemistry. Fischer's subsequent researches, and the researches of a host of carbohydrate chemists in later years, have verified the Van't Hoff-LeBel theory by literally hundreds of accurate tests, without the finding of a single exception. Indeed, if at this time an exception to the theory were claimed, it is most probable that chemists would suspect an error in the experimental work. Van't Hoff, and independently Le Bel, gave us the concept of the asymmetric carbon atom; Fischer used it as a guide for the planning of his experimental work and for the interpretation and systematization of the results.

A detailed survey of the vast number of researches which Fischer published is naturally beyond the scope of this paper; the naming of general fields of work will surely suffice to refresh our recollections of his services to chemistry. In addition to his work in carbohydrate chemistry, his researches in the purine group, his studies of amino acids, polypeptides, proteins, and tannins, and his advancement of the knowledge of enzymes and their specificity, cause him to be regarded as one of the founders of modern biochemistry. In the field of general synthetic organic chemistry mention is to be made of his early discovery of phenylhydrazine and his studies in the indole and the rosaniline groups, his researches on the Heintz acetone bases, on acyl migrations and Walden inversions, and of his syntheses of barbiturates, an accomplishment of enormous practical benefit to mankind in a medical way.

Fischer's experimental discovery of what are now termed the methyl α- and β-glucosides is of particular interest to carbohydrate chemists because he thereby gave the basis for our present knowledge that most carbohydrates have ring-type structures. His later discovery of another methyl glucoside, now known to be a mixture containing principally the methyl α- and β-glucofuranosides, is another of his fundamental contributions. His discovery of the glycal group of carbohydrates, near the end of his life, was a fitting last contribution of prime significance from this master of chemical science.

Many of the names that are in common use in carbohydrate chemistry were introduced by Fischer because of the need for them in the description of his experimental advances; mention may be made of the terms triose, tetrose, pentose, hexose, heptose, etc., of aldose and ketose, and of the specific terms mannose, gulose, idose, talose, ribose, lyxose, threose, and erythrose. Fischer changed the name "dextrose" back to the older name "glucose," that had been suggested by Dumas, because he realized that an "*L*" configurational prefix would be patently unsuitable for the enantiomorph of "dextrose"; the change has received universal approval in scientific writings.

Although Van't Hoff's theory (or principle) of the asymmetric carbon atom is the most easily recognized contribution that he made to carbohydrate chemistry, there are additional subjects in which his creative genius has aided the development of this branch of chemistry.

His osmotic theory of solutions, which extended thermodynamic theory to the study of solutions, particularly of solutions of nonelectrolytes like sugars, has supplied a firm theoretical foundation for the measurement of molecular weights by the routine method of freezing-point depression or of direct observation of osmotic pressure lowering. His studies in chemical dynamics aided the development of this field, which became of much importance in the elucidation of the mutarotation of sugars, and the recognition that these mutarotations are examples of the so-called "balanced" or reversible type of reactions that were studied by Van't Hoff. He also contributed experimentally to the study of the rates and equilibria of reactions that are catalyzed by enzymes, particularly by β-glucosidase; these experiments are among the last of his contributions and show his belief that enzyme actions can be shown to follow the fundamental laws of mass-action. His numerous experimental studies of crystallizations by application of the phase rule of Gibbs clarified the subject of "transition-temperatures" (to use the name that Van't Hoff introduced); his work furnished models for the later measurements by others that constitute the present phase rule diagrams for the solubility and crystallization of such sugars as lactose, maltose, and glucose.

Van't Hoff's service to carbohydrate chemistry was primarily in the field of theory rather than in that of experiment, but there is one instance of such an experimental contribution. In supplement to Pasteur's famous experimental separation of sodium ammonium racemate into a mechanical mixture of crystals of the enantiomorphous forms of sodium ammonium tartrate, Van't Hoff clarified the subject several decades later by determining the phase diagram of this system, through which measurements the "transition-temperature" of 27° became recognized as one of the fixed points of intersection of solubility curves in this system

THE FISCHER INDOLE SYNTHESIS[1]

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THE early history of organic compounds can often be traced to interest in their naturally occurring derivatives. It is of no surprise, therefore, that indole has been extensively studied in world-wide laboratories for almost a century. Its wide occurrence in important biological and chemical systems has provided the stimulus which has resulted in many hundreds of publications devoted to a study of indole and its derivatives. The ancient dye, indigo, the essential amino acid, tryptophan, the plant hormone, heteroauxin, and the recently discovered powerful vasoconstrictor, serotonin, are prominent examples of naturally occurring substances which possess the indole nucleus as a parent. A number of important alkaloids have been shown to be derivatives of indole. Indole itself has been isolated from certain plant sources, notably the jasmines and certain citrus fruits, but by methods which suggest that the indole is actually the degradation product of some higher derivative. It should also be noted that Runge isolated indole from coal tar.

DISCOVERY OF INDOLE

Indole was discovered by Adolph Baeyer as an outgrowth of his famous studies on the constitution of indigo. During the course of his investigation of indigo, Baeyer had at his disposal two indigo derivatives —isatin, its oxidation product, and indigo white, its reduction product. His decision to concentrate his attention on the former is perhaps an example of "chemical intuition," for it led directly to the structure of indole and ultimately to that of indigo. Isatin had been known since 1841, when it was prepared by the oxidation of indigo independently by Erdmann[3] and by Laurent,[4] but its chemical constitution was still unknown. Baeyer and his student, Knop, elected to reduce isatin, and under the conditions they employed obtained two reduction products, C_8H_7NO and $C_8H_7NO_2$, which they regarded as oxygen derivatives of a hypothetical parent, C_8H_7N.[5] The latter substance they named indole. The C_8H_7NO and $C_8H_7NO_2$ compounds therefore became oxindole and dioxindole respectively.

In a second publication later the same year,[6] Baeyer was successful in a complete reduction of oxindole to indole, which he accomplished by distilling a mixture of oxindole and zinc dust. This reaction was noteworthy in a second sense, because it represented the first recorded application of zinc pyrolysis to the reduction of organic compounds. Interestingly enough, this reductive technique was directly responsible for the subsequent confirmation of the structure of alizarin by Graebe and Liebermann,[7] "an achievement which was always a source of great satisfaction and delight to Baeyer."[8]

The structure of indole was finally confirmed by Baeyer and his student Emmerling,[9] who prepared the substance by the fusion of *o*-nitrocinnamic acid with iron filings and alkali; reduction of the nitro group and ring closure to indole resulted. From this reaction, and by consideration of Kekulé's new benzene structure theory, Baeyer assigned it the benzopyrrole structure which soon came into general acceptance.

LACK OF INTEREST

The decade following the discovery of indole saw only moderate interest evinced in its chemistry. At the close of this period Baeyer published two papers dealing with the properties of isatin and two new methods for the preparation of indole,[10] but outside of Baeyer's laboratory the subject was for the most part neglected. Until 1878 Baeyer purposely neglected any further pursuit of the problem out of a misplaced consideration for Kekulé, who had announced his intention to begin an experimental study of isatin. The lack of major interest outside of Baeyer's laboratories may probably be traced to two reasons. Indigo, the derivative which had caused Baeyer to initiate his study, was still a chemical mystery, although Baeyer had conceived, but not proved, its correct structure. After many centuries of speculation and study, however, its drama was nearly ended, and it is not unexpected that indole would be temporarily neglected in favor of the final solution of its more glamorous offspring. A second factor which may have contributed to the neglect of indole was the difficulty faced by experimenters in preparing or obtaining

[1] October 9, 1952, marked the centenary of the birth of (Hermann) Emil Fischer (1852–1919). This paper in a small way may serve as a tribute to the memory of this great organic chemist.

[2] Present address: Organic Chemicals Department, E. I. du Pont de Nemours & Co., Inc., P. O. Box 525, Wilmington 99, Delaware.

[3] ERDMANN A., *J. prakt. Chem.*, **24**, 1 (1841).

[4] LAURENT, A., *Ann. Chim. Phys.*, **3**, 371 (1841); *J. prakt. Chem.*, **25**, 430 (1842).

[5] BAEYER, A., AND C. A. KNOP, *Ann.*, **140**, 1 (1866).

[6] BAEYER, A., *Ann.*, **140**, 295 (1866).

[7] GRAEBE, C., AND C. LIEBERMANN, *Ber.*, **2**, 14, 332 (1869).

[8] PERKIN, W. H., Adolph Baeyer Memorial Lecture, *J. Chem. Soc.*, **1923**, 1520.

[9] BAEYER, A., AND A. EMMERLING, *Ber.*, **2**, 679 (1869).

[10] BAEYER, A., AND H. CARO, *Ber.* **10**, 1262 (1877); BAEYER, A., *ibid.*, **12**, 456 (1879).

workable samples of the compound. Baeyer's route through indigo gave very unsatisfactory yields; his subsequent syntheses through ring closure of *o*-nitrocinnamic acid or N-alkyl-*o*-toluidines were of little more preparative value. Mohlau introduced a new synthesis of indole during this period,[11] but the method as reported at that time yielded only substituted indoles and was of limited practical value. It remained for Emil Fischer to provide a workable laboratory synthesis of indole and its derivatives, and thereby to open up the field of indole chemistry.

FISCHER'S WORK

After receiving his Ph. D. degree with Baeyer in 1874, Fischer remained with Baeyer in Strassburg as an independent research student. In the fall of 1874 the assistant in charge of the organic division left his post, and, at Baeyer's urging, Fischer accepted an appointment to that position. In this capacity he was called upon to assist students who encountered difficulty in their organic preparations. One student, who was assigned the preparation of diphenol by the diazotization of benzidine, regularly obtained muddy products. Not trusting the student's skill, Fischer undertook the preparation himself. Deciding that the disagreeable products were due to oxidation by the nitrous acid, Fischer added to the reaction sodium sulfite to prevent the presumable oxidation. Upon addition of the sodium sulfite he observed the formation of a yellow precipitate. Further experiments revealed the precipitate to be a salt of a phenylhydrazine and resulted in Fischer's discovery of phenylhydrazine itself, published in 1875.[12,13]

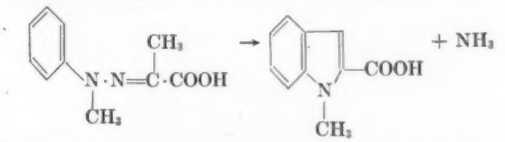
Fischer continued to investigate the preparation and properties of various phenylhydrazines. Although he quickly recognized the ability of aldehydes to combine with hydrazines, he does not appear to have appreciated the enormous value of the reaction as a route to the characterization of carbonyl compounds until 1883. In that year he prepared the phenylhydrazone and methylphenylhydrazone of pyruvic acid. These preparations proceeded with ease and with the production of such beautiful crystalline products that Fischer was encouraged to examine them more closely.

Working with his pupil, Jourdan, on the α-methyl-α-phenylhydrazone of pyruvic acid, Fischer warmed the hydrazone with dilute aqueous hydrochloric acid. Upon examination of the product he noted that the original hydrazone had disappeared and there appeared as a product a new carboxylic acid of unknown constitution. With characteristic thoroughness he analyzed the substance and recognized that the following equation could account for its molecular formula.[14]

$$C_{10}H_{12}N_2O_2 \rightarrow C_{10}H_9NO_2 + NH_3$$

Fischer admitted that he could neither identify the product nor account for its formation, saying: "The foregoing (reaction) is so remarkable that we meanwhile do not hazard to give an explanation for it. In any case, the new acid is representative of a remarkable class of substances, for which analogies until now are absent."[14]

The publication of these results was succeeded by a paper the following year by Fischer and Hess, in which they identified the reaction as producing a derivative of indole according to the reaction below.[15]



The yield of the 1-methylindole-2-carboxylic acid prepared in this manner was only about five per cent, a yield so low that a less capable experimenter might have missed it entirely.[16] That Fischer recognized the potential value of the reaction, however, is clear from his statement that "For the synthesis of compounds of the indigo group a new and, as it appears, quite fertile territory is opened."[15]

Fischer appreciated the reaction as a convenient route to indole derivatives of many types. Accordingly, he continued his investigations, publishing his results in the *Berichte* and the *Annalen* over the period of the next four years. His further researches led him to a number of refinements of the original procedure. He found, for example, that the original conversion of the methylphenylhydrazone of pyruvic acid into its corresponding indole derivative proceeded more smoothly and in better yield if anhydrous zinc chloride was substituted for hydrochloric acid as the catalyst.[17] His modified procedure utilized a three to five molar excess of zinc chloride and heating in the neighborhood of 200°C. Using this technique, for example, Fischer was able to convert propionaldehyde phenylhydrazone to skatole (3-methylindole) and acetone phenylhydrazone to 2-methylindole with respective yields of 60 and 35 per cent.[18]

The announcement of these results undoubtedly served to stimulate interest in the chemistry of indole. A convenient route to indoles of widely different structures was made available, and one of the obstacles to the investigation of indole was largely eliminated. Conditions and catalysts have since been altered and improved, but the cyclization of a phenylhydrazone to an indole is still correctly referred to as the Fischer indole synthesis.

[11] MOHLAU, G., *Ber.*, **14**, 171 (1881).

[12] FISCHER, EMIL, *Ber.*, **8**, 589 (1875).

[13] The account of the discovery of phenylhydrazine is taken from: FISCHER, EMIL, "Aus Meinem Leben," Springer-Verlag, Berlin, **1922**, p. 52.

[14] FISCHER, EMIL, AND F. JOURDAN, *Ber.*, **16**, 2241 (1883).

[15] FISCHER, EMIL, AND V. F. HESS, *Ber.*, **17**, 559 (1884).

[16] This reaction has been repeated by the author several times in the laboratories of Tulane University with yields of 71 to 78 per cent. ROUSSEL, P. A., "The metalation of indole and pyrrole and some of their N-substituted homologs with *n*-butyllithium," dissertation, Howard-Tilton Memorial Library, Tulane University, New Orleans, La.

[17] FISCHER, EMIL, *Ber.*, **19**, 1563 (1886).

[18] FISCHER, EMIL, *Ann.*, **236**, 116 (1886).

Emil Fischer

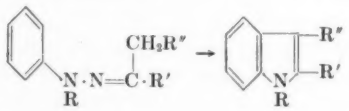
VERSATILITY OF SYNTHESIS

Modern applications of the Fischer indole reaction have resulted in a number of refinements and modifications. The use of large excesses of zinc chloride is now recognized as unnecessary, catalytic amounts being capable of causing reaction. A large number of new catalysts have been introduced, their efficiencies depending on the specific reaction they are called upon to catalyze. Included in the list of recognized catalysts are such diverse materials as hydrochloric, sulfuric, and acetic acids, zinc chloride, cuprous chloride, cobaltous chloride, nickelous chloride and the recently discovered catalysts stannous chloride, boron trifluoride, and the Grignard reagent.

Other improvements in Fischer's early process include the substitution of inert solvents for the aqueous or fused media employed by Fischer. Methylnapthalene has been observed to serve efficiently in this respect.

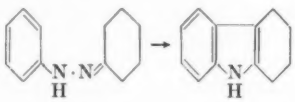
A survey of the literature regarding Fischer's indole reaction reveals that it is indisputably the most versatile of all laboratory indole preparations. The synthesis permits a wide latitude of changes in both the phenylhydrazine and the aldehyde or ketone whose hydrazone it forms. The ease with which the reaction proceeds is largely governed by the nature of the phenylhydrazine and the carbonyl compound. Some reactions are remarkably easy to bring about, *viz.*, the preparation of 1-methylindole-2-carboxylic acid from the methylphenylhydrazone of pyruvic acid merely by warming with dilute hydrochloric acid. The extreme of difficulty is represented by the transformation of propionaldehyde phenylhydrazone into skatole (3-methylindole) which requires a temperature of about 200°C. As a general rule the ring closures to indoles are brought about by conditions within a smaller region lying between these two extremes, N,N- disubstituted phenylhydrazones usually reacting with greater ease than their unsubstituted homologues.

The extensive application of his indole synthesis as a laboratory route to substituted indoles has only corroborated Fischer's early prediction of the usefulness of the method. So few exceptions to the versatility of the reaction have appeared that it becomes possible to write for it a general equation:



Although there are obvious steric requirements, the R groups may in general be hydrogen or any species of hydrocarbon radical.

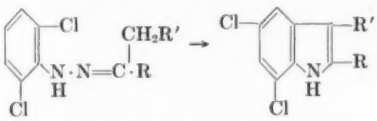
The wide applicability of the Fischer indole synthesis to the preparation of indoles is illustrated in the examples following. Ketones ranging in complexity from acetone to succinaldehydic acid form phenylhydrazones which readily give the corresponding indole derivatives. Phenylhydrazones derived from cyclic ketones provide interesting evidence of the versatility of the Fischer synthesis. The phenylhydrazone of cyclohexanone, for example, yields a partially reduced carbazole with great ease.



The Fischer synthesis also permits wide variation in the character of the phenylhydrazine. It has already been noted that N,N- disubstituted phenylhydrazones are more readily transformed into indoles than unsubstituted phenylhydrazones. Similarly, the presence of substitutents in the phenyl nucleus of the phenylhydrazone does not in general reduce its ability to undergo ring closure. Bromo-, methyl-, and even such strongly deactivating groups as nitro- do not preclude reaction. Indeed, even the presence of substituents in both of the positions *ortho* to the hydrazine group does not always prevent ring closure. It has recently been demonstrated that such a group may migrate to another position in the molecule in order to allow cyclization to occur in the positions *ortho* to the hydrazine nitrogen.[19] An ex-

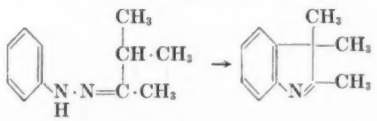
[19] CARLIN, R. B., J. G. WALLACE, AND E. E. FISHER, *J. Am. Chem. Soc.*, **74**, 990 (1952).

ample of such a reaction is illustrated in the equation below.



As a general rule in substituted phenylhydrazones, ring closure is directed to the carbon atom *ortho* or *para* to the substituent, regardless of its nature.

An interesting extension of the Fischer indole synthesis which further testifies to its utility is found in the reactions of phenylhydrazones derived from ketones which possess no *alpha* methylene group. Such a case is found in the phenylhydrazone of methyl isopropyl ketone, which reacts within the framework of the Fischer synthesis to yield a substituted indolenine, indolenine being a tautomer of indole. One such reaction, discovered by Plancher in 1898,[20] is illustrated below.

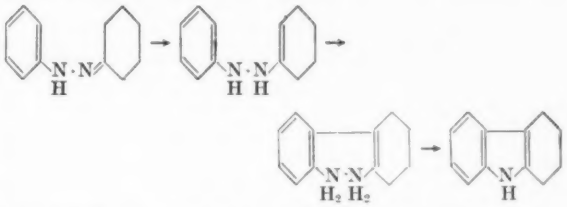


Despite this summary of reactions which illustrates the wide scope of the reaction, certain exceptions to the generality of the Fischer indole synthesis are recognized. Perhaps the most significant of these is the failure of acetaldehyde phenylhydrazone to yield indole itself. The other important exception is noted in the phenylhydrazones of β-keto esters, which usually yield substituted pyrazolones rather than indoles.

MECHANISM

A great deal of speculation has appeared in the literature regarding the mechanism of the Fischer indole reaction. Fischer wrote the equation for the reaction as proceeding through the elimination of a molecule of ammonia from the phenylhydrazone. In his reactions involving the ring closure of N,N- disubstituted hydrazones both of the groups attached to the hydrazine nitrogen remained in the indole which was ultimately formed, and Fischer considered this evidence that it was the other nitrogen atom which was lost as ammonia (ref. 15). Recent advances in the use of isotopes have enabled modern chemists to examine this proposal. Allen and Wilson investigated the Fischer indole synthesis using phenylhydrazones bearing N^{15} in the hydrazone group and as a result of their studies offered conclusive evidence that it is the nitrogen more distant from the phenyl group which is lost as ammonia.[21] Fischer's postulation was therefore confirmed.

Perhaps the most tenable mechanism which has been advanced to explain the Fischer indole synthesis has been that of Robinson and Robinson, which first appeared in 1918.[22] Although the Robinsons' mechanism does not include electronic structures or interpretations, each postulated step is in accord with reactions which are known to occur in analogous cases. The cyclization of the phenylhydrazone of cyclohexanone provides an illustration of this mechanism.



The initial step involves the shift of a proton in the hydrazone to form a substituted hydrazine, which is tautomeric with the hydrazone. The second step involves an *o*-benzidine rearrangement, followed in the third step by the elimination of a molecule of ammonia as a salt of the catalyst. Strong experimental support can be cited for each of these postulated steps. The last decade has seen the appearance of a number of refinements of the Robinsons' mechanism, interpreting it in terms of modern electronic theory. These publications, notably those of Carlin,[23] considerably strengthen the mechanism of the Robinsons, though by no means do they exclude further argument.

Although the Robinson and Robinson mechanism seems to provide an adequate explanation for the Fischer indole synthesis, certain other mechanisms have appeared which should not be excluded from consideration. Reddelien,[24] Bamberger,[25] and an English school led by Pausacker[26] have advanced mechanisms which have certain strong points in their favor.

Modern organic chemistry is indeed indebted to Emil Fischer for the indole synthesis that has come to bear his name. What began as a study of the reactions of phenylhydrazine led a remarkable chemist to a remarkable reaction, a discovery that has contributed immeasurably to the development of the chemistry of indole and of its natural products.

ACKNOWLEDGMENT

The author is indebted to Dr. Clara deMilt for her invaluable assistance in the preparation and correction of this manuscript.

LITERATURE

For general reviews of the chemistry of indole the reader is directed to:

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Elderfield, R. C., Editor, "Heterocyclic Compounds," John Wiley & Sons, Inc., New York, **1952**, Vol. III, pp. 1–275.

[20] Plancher, G., *Ber.*, **31**, 1496 (1898).

[21] Allen, C. F. H., and C. V. Wilson, *J. Am. Chem. Soc.*, **65**, 611 (1943).

[22] Robinson, G. M., and R. Robinson, *J. Chem. Soc.*, **113**, 639 (1918); *ibid.*, **125**, 827 (1924).

[23] Carlin, R. B., *J. Am. Chem. Soc.*, **74**, 1077 (1952).

[24] Reddelien, G., *Ann.*, **388**, 179 (1912).

[25] Bamberger, E., and A. Landau, *Ber.*, **52**, 1097 (1919).

[26] Barnes, C. S., K.H. Pausacker, and C. I. Schubert, *J. Chem. Soc.*, **1949**, 1381.

THE EVALUATION OF MOLECULAR FORMULAS

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IN QUALITATIVE organic chemistry courses, a common exercise is the solution of problems in which the molecular formula of a compound is given, and some of its properties and reactions. It is then required that the structure of the substance in question be deduced. Frequently one finds that students have difficulty in evaluating the molecular formula in terms of the amount of unsaturation present. In this note the concept of the "equivalent hydrocarbon" is presented as a useful teaching device, which enables the student to obtain the maximum information from the data, and also to have a useful starting point for the application of the qualitative reaction results which are given. This method has been used successfully for five years by students at Melbourne Technical College, and more recently, in qualitative organic courses at Notre Dame. With a little practice in its application students find that they save time in evaluating formulas, and they approach the solution of such problems with greater confidence.

The equivalent hydrocarbon is defined as that compound containing only carbon and hydrogen which is equivalent to the compound in question in the sense that *it contains the same number of rings and multiple bonds*, but not necessarily the same number of carbon atoms. By comparing the equivalent hydrocarbon with the corresponding saturated aliphatic hydrocarbon, the number of rings and multiple bonds can be obtained directly. The principle involved is simply that of homologous series, as can be seen from the following rules and examples.

Rule 1. To obtain the equivalent hydrocarbon from the molecular formula of an organic molecule which is not a salt, (i) replace each oxygen and sulfur (or other Group VI atom) by CH_2, (ii) replace each nitrogen (or other Group V atom) by CH, (iii) replace each halogen atom by hydrogen.

The molecular formula has now been reduced to one containing carbon and hydrogen only, *and must have an even number of hydrogen atoms*, C_nH_{2m}. This last requirement provides a useful arithmetical check. Now determine the formula of the corresponding saturated aliphatic hydrocarbon, C_nH_{2n+2}. Subtract the first formula from the second: The difference is an even number of hydrogen atoms. One-half of this number $(n-m+1)$ is the number of rings and multiple bonds in the original molecule. No distinction is made between the different kinds of multiple bond (*e. g.*, C=C, C=O, C=N, N=N, etc., or C≡C, C≡N), but the sulfur-oxygen and phosphorus-oxygen coordination covalences appear as single bonds. No distinction is made either between the different sizes of ring, or whether a ring is homo- or heterocyclic. At this point the qualitative data must be used to account for the observed number.

Rule 2. To obtain the equivalent hydrocarbon from the molecular formula of a salt: (i) in the case of inorganic salts of organic bases, subtract the formula of the acid from that of the salt before proceeding as described above. When dealing with salts of dibasic acids, the result of the subtraction must be halved; (ii) in the case of metal salts of monobasic organic acids, replace the metal ion by the same number of hydrogen atoms as its valence and divide the total by the valence. The extension of the method to salts of polybasic organic acids presents some difficulties.

The method is particularly advantageous in problems where a series of compounds is converted one into another, and it then becomes a useful supplement to the techniques described in such texts as Shriner and Fuson.[2]

EXAMPLES

(*a*) *Molecular Formula* C_4H_6. There are no atoms other than C and H. The corresponding saturated aliphatic hydrocarbon is C_4H_{10}. The difference is four hydrogen atoms and one-half of this is two. This molecule contains therefore two rings, or one ring and one double bond, or two double bonds, or one triple bond. The qualitative data should enable the student to choose between these possibilities, cyclobutene, butadiene, and the butynes being the obvious ones. The equivalence of a triple bond to two double bonds should not require elaboration.

(*b*) *Molecular Formula* C_4H_8O. Replacing the oxygen atom by CH_2, we find the equivalent hydrocarbon to be C_5H_{10}. The corresponding saturated aliphatic is C_5H_{12}. The compound therefore contains one ring or one double bond. Cyclobutanol, methyl ethyl ketone, butyraldehyde, and ethyl vinyl ether fit the quantitative data, as well as three-membered ring systems such as the epoxybutanes and cyclopropylcarbinol, and the five-membered heterocycle tetrahydrofuran.

(*c*) *Molecular Formula* C_6H_6 (*Benzene*). Application of the rule indicates that benzene has four rings or multiple bonds. In this and other aromatic systems, the Kekulé formula is adequate for our purpose: the benzene molecule has one ring and three double bonds.

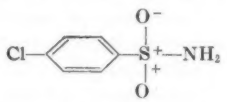
[1] Present address: University of Notre Dame, Notre Dame, Indiana.

[2] SHRINER, R. L., AND R. C. FUSON, "The Systematic Identification of Organic Compounds," 3rd ed., John Wiley & Sons, Inc., New York, 1948, Chap. XI.

(*d*) *Molecular Formula* $C_6H_4NO_2Cl$ (*p-Nitrochlorobenzene*). From the computation one finds the equivalent hydrocarbon to be C_9H_{10}, so that the molecule has

five rings or multiple bonds. Notice that the nitro group has *one* double bond. Note also that the procedure fails for halogen compounds in which anomalous valencies occur, *e. g.*, iodobenzenechloride, $C_6H_5ICl_2$, in which iodine is three-covalent.

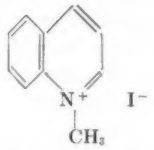
(*e*) *Molecular Formula* $C_6H_6NO_2SCl$ (*p-Chlorobenzenesulfonamide*). Replacing oxygen and sulfur by CH_2, nitrogen by CH, and chlorine by H, the equivalent hydrocarbon is found to be $C_{10}H_{14}$; the corresponding saturated aliphatic is $C_{10}H_{22}$, whence the number of rings and double bonds is four, which are all accounted for in the benzene ring. Once again it is emphasized



that the sulfur-oxygen bond in sulfonic acids, sulfonamides, sulfoxides, sulfones, and similar compounds are counted as single bonds, whatever their true structure may be.

(*f*) *Molecular Formula* $C_4H_6O_4Ca$ (*Calcium Acetate*). The equivalent hydrocarbon is $(C_8H_{14}+2H)/2 = C_4H_8$, whence it appears there is one double bond in each organic unit, the C=O in the carboxylic acid group.

(*g*) *Molecular Formula* $C_{10}H_{10}NI$ (*Quinoline Methiodide*). Subtracting the formula of the acid, HI, we have $C_{10}H_9N$, for which the equivalent hydrocarbon is

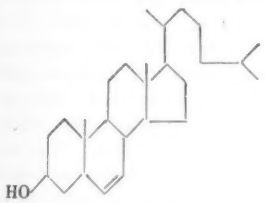


$C_{11}H_{10}$. It follows that the molecule contains seven rings and multiple bonds, as in the Kekulé formula. In such cases one must know from the qualitative data that a salt is under consideration.

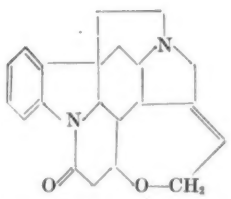
Finally, four more or less complex naturally occurring substances are considered below to emphasize the generality of the method.

ACKNOWLEDGMENT

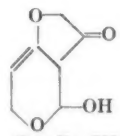
The author is indebted to Dr. E. L. Eliel and Dr. G. F. Hennion for constructive criticism during the preparation of this paper.



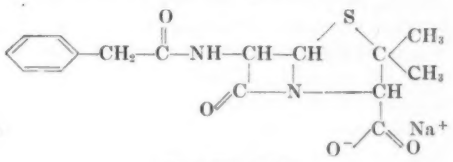
Cholesterol, I



Strychnine, II



Patulin, III



Penicillin G, IV

Substance	*Molecular formula*	*Equivalent hydrocarbon*	*Rings and multiple bonds found*	*Rings and multiple bonds present*
I	$C_{27}H_{46}O$	$C_{28}H_{48}$	5	4 rings 1 C=C
II	$C_{21}H_{22}N_2O_2$	$C_{25}H_{28}$	12	7 rings 1 C=C 3 C=C aromatic 1 C=O
III	$C_7H_8O_4$	$C_{11}H_{16}$	4	2 rings 1 C=C 1 C=O
IV	$C_{16}H_{17}N_2O_4SNa$	$C_{23}H_{30}$	9	3 rings 3 C=O 3 C=C aromatic

ANALYZING STRAIGHT LINE DATA[1]

FORMAN S. ACTON
National Bureau of Standards, Los Angeles, California

WHEN an engineer cautiously approaches a statistician, graph in hand, and asks how he should fit a straight line to these points, the situation is not unlike that moment when one's daughter inquires where babies come from. There is need for tact, there is need for delicacy, but here is opportunity for enlightenment and it must not be discarded casually—or destroyed with the glib answer.

Here is a man with a problem. He has asked a simple question to which there is no answer, and the statistician must take it upon himself to help find out the real questions to which answers are important, pertinent, and possible.

Why do people keep fitting straight lines to data? What is the fascination they hold for the experimental man? It certainly is not the aesthetic division of a plane into two halves—the delight of the geometer and analyst. The biometrician may wish to separate sheep from goats, but the chemist is happiest if his points fall on this boundary and separation is his farthest thought. Perhaps the line is valued because it is a simple two parameter curve—but then the sine wave is equally valuable—and any horizontal line becomes more important than either because it is simpler and possesses only one parameter. I think that for most physicists, chemists, and engineers, the straight line is valued chiefly because it can be extrapolated. Certainly no other curve can claim this property with the same degree of psychological confidence. A man feels much surer when he extrapolates a line than when he extrapolates any other curve—and in this day of bigger operations and larger productions, the engineer must extrapolate. The atomic bomb could not have been built without the straight line and econometricians in the Bureau of the Budget doubtless find it equally indispensable.

Extrapolation is merely one aspect of *prediction*, and here is one of the two important reasons that we fit curves to data. We desire to summarize our experimental experience in the form which may be used to predict the outcome of future similar experiments. This purpose the physical scientist shares with the biological and social scientists. But he differs from them in that he has another purpose more important than prediction: *the estimation of structure*. Our physical sciences have carefully chosen their definitions so as to invoke linear, or nearly linear relations between the various observable quantities, or simple transformations of these quantities. (If exponential behavior is expected, the logarithms are plotted, if pressure and temperature are examined, the reciprocal of the temperature is used; if Boyle's law is violated at heavy pressures, correction coefficients are introduced. By these artifices we retain the intuitional and extrapolatory advantages of the straight line for our machinations, and the statistician reaps the benefit in the simplicities of linear models.) These linearized physical hypotheses or laws contain structural parameters which the experimenter is trying to estimate. Perhaps it is the resistance of a piece of wire, to be found by applying measured voltages and recording the corresponding current; again it may be the angle of optical rotation of a chemical in solution as a function of its concentration, the temperature of the system, and wave length of the light. The system may be simple or complicated, but a theory specifies a functional form containing parameters and the experimental data are used to estimate these parameters. Ultimately we may wish to use this law for prediction, but frequently we merely wish to test our hypothesis that the exponent in the resistance law is *one* (*i. e.*, the current is a linear function of voltage), or some such simple value which the theory hopefully predicts. The experimenter's data contain errors which he probably regards as nuisances to be averaged out or eliminated by fitting a line—a line which is best in some sense. The only quantities of real interest to him are the slope and the intercept of this best line. This is the philosophy which bred the cult of Least Squares, and it is here that a guiding hand can effect the greatest good by pointing out other approaches to the same data which are apt to be more profitable.

Let us take an example. Suppose a physicist is trying to measure the effect of temperature on the fluorescence of a phosphor under a standard intensity of ultraviolet light. He has measured the intensity of the fluorescence while holding the phosphor temperature constant at each of several levels. The data, temperature versus intensity, are not really linear, and they scatter quite a bit. Perhaps a plot of intensity (or square root of intensity) versus the reciprocal of absolute temperature will linearize the data and perhaps it will not, according to the physical law involved, but the scatter has to be dealt with if any precision is to be obtained. The scatter arises in part from errors in measurement, but also from the fact that the light source is not constant and from the aging of the phosphor with oxidation by air or with exposure to ultra-

[1] The preparation of this paper was sponsored (in part) by the Office of Naval Research. It was presented at the annual meeting of The Institute for Mathematical Statistics, December 28, 1951.

violet light. The various errors introduced by the measuring instruments and the operator are lumped together.

I shall not attempt to be completely realistic here, but, if the intensity of the incident ultraviolet radiation is recorded, the uncontrollable fluctuations in this source may well cause enough variation in the final fluorescence to permit at least a crude estimation of the effect of intensity, and hence to permit its removal from the structural picture. If our physical friend's purpose is to estimate the parameters in the equation connecting intensity of fluorescence with temperature, then allowance for this residual variaton in the light source should greatly increase the precision of his estimates. If, on the other hand, his purpose is to calibrate this particular piece of phosphor so that he can use it as a thermometer in some inaccessible place, then the fluctuations of the light source are even more important, because they will also occur in the later experiments. Here he is interested in the *prediction* problem, and the effect of variations in the light source must be estimated in order to be allowed for realistically in the final apparatus. Undoubtedly a different source will be used later, and since its variability will be different from the one used in calibration, an estimation of the magnitude of this effect is essential. (Whether or not this estimation of intensity effects can be made here is dependent on the time constants of the phosphor as compared with the "period" of the light source fluctuations. If the phosphor responds much faster than the light varies, then the estimation of this effect is easy. If the phosphor is sluggish, then it will give an average effect over time, making its estimation difficult, and requiring subsidiary direct experiments on intensity if allowance is to be made for these fluctuations.)

The effect of aging in the phosphor requires different treatment. This is really another structural estimation problem, and hence requires a structure—a theory to give the functional form. If the effect were expected to be a linear decrease in the entire activity level with time, then the rate of decrease could be found by a series of experiments over several weeks at one temperature level. On the other hand, if the effect of time were expected to change the sensitivity of the phosphor to temperature, then a more elaborate investigation would be required, involving several levels of temperature at each of several times, the exact design of the experiment and analysis of the data depending rather strongly on the functional forms contained in the underlying physical theory.

In both the structure and the prediction problem, we are interested not only in fitting a line, but also in estimating the variation in the data which is not explained by the line, and especially in breaking that variation up into as many assignable components as seem justified by the theory and data available. The more complete the breakdown, the more thoroughly we shall understand what is really going on, and the better we will be able to cope with the prediction problems into which these components of variance must inevitably enter.

MATHEMATICAL MODELS

To break down the variability of our data into its smallest possible classifications, we must have a reasonable mathematical model and our algebraic techniques will depend directly on what particular model we adopt. I propose to discuss some of the models which seem to occur in the physical sciences with some frequency in order to emphasize their variety, if not to offer a pat formula for treating each one in detail. The treatment depends, as we have already said, not only on the model but also on the particular data, so detailed discussion of some points is possible only when we are confronted with data from a real experiment, and faced with the necessity of answering pertinent questions. This takes more space than is available, and I shall limit my discussion to the simpler models, showing, where possible, the sorts of questions which might naturally be asked.

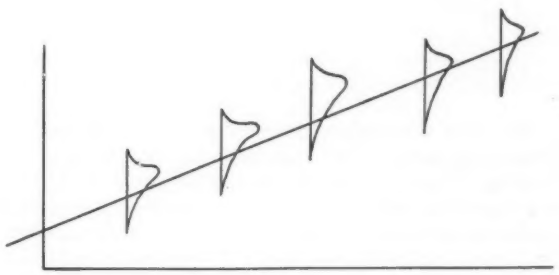


Figure 1

x without error. The most commonly discussed model involving a straight line is the one in which one variable is measured "without error," while the other is assumed to exhibit all the variability. This is the classical case treated by Gauss, known to physicists, and used by everybody else. If we sample repeatedly at one fixed value of x, we obtain a distribution of y values which is shown pictorially in Figure 1. The distributions are sketched in perspective, and—if we are lucky—look somewhat like the famous normal one. If we are still luckier, all of these distributions are identical, merely being located at different places near the line. Fitting a line to this underlying model is the ancient and honorable application of Least Squares, but the extraction of information about the residual variability goes far beyond such a mechanistic attitude. Later we shall have more to say about even this well-known model.

x and y both in error. When we consider that x and y are both measured erroneously, we face much more complicated situations. Mathematically simplest among these is the one espoused of late by Berkson where x is "set" at predetermined values, which are recorded, and y is then measured. Actually the errors in x do not enter into the numbers, X, recorded; they are uniformly and unimaginatively the same—being

the values to which the experimenter fondly hopes he has set his instrument. Because of this, the situation is actually one-variate-in-error, and, although we may admit that x is not precisely known, there is nothing the statistician can do about it, since the information is not present in the data. The graph displays the measured variable y and the idealized variable X, and thus the line shows the relation between these two. The real x is never measured. (Two centuries ago Bishop Berkley proclaimed a philosophy which denied the existence of an oak tree when no one bothered to observe it; two years ago Physician Berkson did the same for errors.)

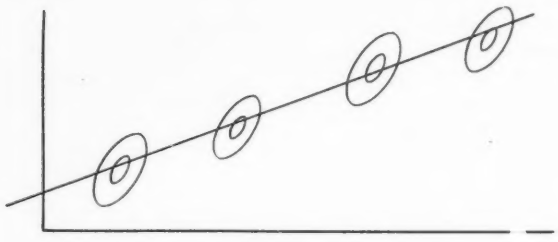


Figure 2

The true two-variates-in-error model hypothesizes a line along which the experimental system may be varied. If we set the system at a particular unknown point on this unknown line and repeatedly measure x and y, we generate a small bivariate distribution (which we hope is sort of normal) and if we move to a new point we generate a new distribution there. If we are complete optimists, we assume that the shapes of these several distributions are identical, differing only in the locations of their means. Like the one dimensional case, they may be located *on* the line, or merely near it. A fairly optimistic picture is shown in Figure 2, where we note that the error distribution contours show the x and y errors to be positively correlated—the ellipses are not parallel to the x and y axes. (They are intended to be geometrically similar, however.)

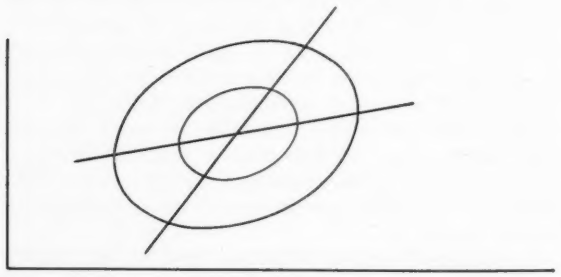


Figure 3

Still another two-variables-in-error model is the single bivariate distribution from which random or stratified samples are drawn—the heights of fathers versus the heights of sons, for example. This is not a particularly common model in the nonbiological sciences and so we introduce it here merely to say that we do not, in this paper, refer more than is absolutely necessary to this situation—one often confused with the structural fitting of interest to chemists and physicists. It is the model which gives rise to the two different lines, y on x and x on y, neither of which is the major axis of the ellipse. Figure 3 shows the intruder, and with this we shall try to dismiss him. Our attempts to get rid of our unwanted guest are foredoomed, however, as a glance at Figure 2 shows his children are already well established in our models there. We therefore make the best of a bad situation by discussing him only when we cannot do otherwise.

TYPES OF VARIABILITY

Whether our interest is structure or prediction, we need to know how the variability enters into our points before we can decide how stable our line may be or how close to it a new point is apt to fall. With the simpler models shown above there are two general sources of variability which can usefully be distinguished: (1) the amount by which points jump around within one of the elementary distributions, and (2) the amount by which the distribution means jump around the best fitted line.

The first of these types of variability is the variance of the residual "error"—it is measured by the Mean Squares Within (the finest subgrouping), to use the terminology of Analysis of Variance. The second type, in physical experiments, is apt to represent the effect of some uncontrolled variable which is nearly constant for each distribution separately, but varies from one to the next; or it may merely reflect the nonlinearity in the system, not accounted for by the theory. If we have more complicated models, with more categories of points (points in distribution I measured on Tuesday, by operator A as opposed to operator B on Wednesday), then we will get more categories of variability, some of which may cause subdivision of the two categories already mentioned, and some of which may occur in new ways—by rotating the error distributions, to mention a horrible example.

A third category of variability, which turns up in all the formalism of Analysis of Variance, is that attributable to the line itself. Since we are usually quite aware of this line, to regard it as a source of variability appears artificial, but it is done for a consistent mathematical treatment and need not trouble those who object to it. Just ignore that particular interpretation.

In order to estimate the first and finest category of variance, the Sums of Squares Within the error distributions, it is clearly necessary to have at least two points from each distribution. We then get independent estimates of the variance, σ_u^2, σ_v^2, and the covariance $\rho\sigma_u\sigma_v$ from each cluster, which may then be pooled into better estimates if we feel justified in assuming the shapes of all of these distributions to be alike. These remarks apply equally to the models with one or both variates in error. Although the SSW cannot generally be estimated by taking only one point from a distri-

bution, in one fairly common one-dimensional model, however, we can apparently estimate the SSW even with one point for each value of x, provided we are willing to assume that all the distributions are identical except for the location of their means and further that these means all fall on a true line (thereby denying the second category of variability mentioned above). In this special case, we fit a best line and then (conceptually) project all our points parallel to the fitted line down onto the y axis. Such a projection piles up all the separate similar distributions on top of one another, giving a single distribution exactly like each one of the originals. We now find ourselves with several points from one distribution and can estimate the variance quite easily. This sum of squares is merely a sum of squared deviations from the line, and is quite familiar to anybody who has fitted the simplest examples. An analogous technique is clearly not available with two dimensional error distributions unless we know where on the line the centers of these distributions are supposed to be—a most unlikely piece of information.

When we compare the no-error-in-x models with the two-variables-in-error ones, we see many similarities, but a few striking differences. In one dimensional error there is little question about which points belong to what distribution. Either we have several y values at a particular value of x, or we do not. The means of these distributions are easily found if there are several points, or it is merely the point itself if only one datum is given. When we examine the two-dimensional picture, we see that if the sample distributions lie close together it may not be at all clear to which one any particular point may belong, unless the experimenter has been able to label them. We do not know whether several points all come from one distribution, each point estimating the same "true point" (the mean of the distribution), or whether each point came from a separate though near-by distribution such as would occur if the true value of either x or y really changed a small amount between observations.

Still a further difficulty besets us when we try to decide how far a distribution mean is displaced from the line. In the one dimensional case the only displacements are vertical, but with two dimensional errors we have to decide just what point on the line is "nearest" the distribution mean, in the sense of being the most likely. Figure 4 shows a hypothetical error distribution and a line, indicating the point which is "nearest in probability." Note that this point depends on both the shape of the distribution and the slope of the line. Thus we have to know something about the general shape of the little error distributions before a line can be fitted and before the variation can be broken down to its component parts. (If we may assume bivariate normal distributions, the contours are ellipses and we must know both their eccentricity and their angle of tilt. This is frequently expressed as the equivalent requirement of knowing ρ_{uv} and σ_v/σ_u.)

If the shape of the individual error distributions is to be found from the SSW (*i. e.*, from the way a point tends to reproduce itself on repeating an experiment), then analogously the slope of the physical law is determined chiefly by the Sums of Squares Between, the amount by which the various clusters are separated in both x and y. The further apart our points, the better the slope can usually be determined. Sometimes however, the spread of our data is not under our control;

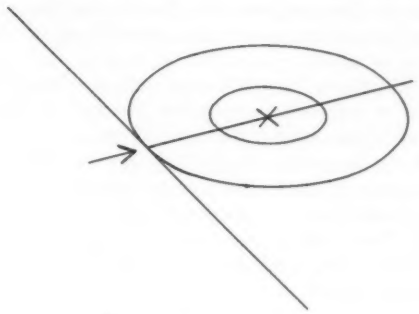


Figure 4

the variables may only be measurable in certain ranges, or perhaps they are not of human origin. In this case the problem arises that in addition to the little distributions we also have a distribution applied to x itself, and hence we can either take all the values we can get—bunched or widely distributed—or we can artificially separate them into several groups, a procedure which is intuitively distasteful.

NUMERICAL EXAMPLE

In order to illustrate some of our points, we take a set of data containing an underlying linear relation between two variables, both of which were repeatedly measured with error. To avoid losing sight of the main argument in a mass of experimental detail, we have manufactured these data to our own specifications. This has the further advantage that we know the underlying model, and can see how thoroughly we recover from the data the information we so carefully put in. The basic error distribution is assumed to be a bivariate normal with $\sigma_x^2 = 0.83$, $\sigma_y^2 = 0.56$, and $\rho\sigma_x\sigma_y = -0.186$. The underlying line is $y = 1.0 + 0.4x$, and ten measurements were made at each of the (true) points $(-4, -0.6)$, $(0, 1.0)$, $(2, 1.8)$, and $(6, 3.40)$. The values obtained are given in the table.

x	y	x	y	x	y	x	y
−5.88	−0.26	−1.62	1.97	1.40	1.65	6.11	3.30
−5.52	−0.99	−0.58	1.16	1.61	1.65	6.74	2.98
−4.59	−1.48	0	1.35	1.72	0.65	6.80	2.90
−3.68	−0.73	0.95	1.90	1.88	2.09	7.63	3.45
−3.76	0.06	1.18	−0.14	4.05	0.20	8.80	3.16
−5.22	−0.27	−0.92	2.15	0.05	3.35	7.27	2.70
−4.10	0.20	0.94	1.46	0.67	2.39	5.30	4.55
−4.87	−0.30	1.47	0.90	0.91	1.98	5.13	4.03
−4.04	−0.70	0.25	0.62	1.10	1.80	4.37	3.65
−3.18	−0.53	0.26	1.15	1.84	1.07	5.00	3.24

If we now pretend that we are a desperate statistician faced with these data and seeking a slope, knowing that a line is contained therein but otherwise unaware of the model, we might choose several courses:

(1) We might rashly decide to take x as accurately measured, ascribe all error to y, assume each error to be normally distributed fit by Least Squares, and set limits on the slope by the standard t-test.

(2) We might assume errors to be present in both x and y, use the maximum likelihood equation to determine a best slope, and wring our hands for lack of confidence limits.

(3) We might use the techniques of components of variance in regression, given by Tukey,[2] to find both a slope and some approximate limits for it.

Since we have no desire to compare the detailed mechanisms of these fitting procedures, we merely give a breakdown of the standard sums of squares and the final slopes as computed by the methods mentioned. The data are presented merely to allow the curious reader to try his own pet schemes for comparison.

Using a computational form analogous to matrix notation, we find the Sums of Squares for the data in the table to be:

Total SS	$\begin{Vmatrix} 635.16168 & 199.06321 \\ & 93.33410 \end{Vmatrix}$	$=$	$\begin{Vmatrix} S_{xx} & S_{xy} \\ & S_{yy} \end{Vmatrix}$
SS between groups	$\begin{Vmatrix} 591.96957 & 213.21946 \\ & 76.82669 \end{Vmatrix}$	with 3 degrees of freedom	
SS within groups	$\begin{Vmatrix} 43.19211 & -14.15625 \\ & 16.50741 \end{Vmatrix}$	with 36 degrees of freedom	

If we treat the x as known accurately, the slope, b, is 0.313 and five per cent limits on each side are 0.241 and 0.386. This incorrect model gives limits which don't even *include* the correct slope.

Maximum likelihood fitting gives a slope of 0.360, which is fairly good.

Regression components lead to 0.364 with 90 per cent confidence limits of 0.291 and 0.430.

If we now discard all our x observations, replacing them with the "true" values -4, 0, 2, 6, we create a Berksonian example which, however, is not comparable with the one above because it utilizes exact knowledge about x. Here the line is y-observed versus x-recorded and hence only y contains errors. The slope of this line is 0.383 with 90 per cent confidence limits of 0.323 and 0.442, given by the classical t ratio.

SOME PHYSICAL SYSTEMS

In order to give physical meaning to the mathematical models we shall mention briefly some experiments, rather idealized to be sure, which might be expected to display some of the types of variation we have been discussing. Simplest among these would be the determination of the elastic constant of a spring which obeyed Hooke's law. Here known weights, x, are applied to the spring and the elongation, y, is measured. The weights are presumed to be known accurately, so we obtain the classical model of error in y only. Repeated measurements with each of several weights would be possible so the assumption that the error distributions were independent of x could be experimentally checked. (If the error turns out to be proportional to the weight, a plot of log y versus log x will restore us to the more pleasant homoscedastic model). If we should perform our Hooke's law experiment using rocks for weights, these rocks then being weighed on kitchen scales to get x, we have a two-error model and again the measurements are repeatable if we think the spring is not going to be deformed by any particular weighing, and individual distributions may be generated. In view of the frequency with which Hooke's law is a bad approximation, the pertinent questions probably include a determination of the slope of the line with confidence limits, inquiring about whether a straight line is justified or whether a parabola is needed, and perhaps even a question about whether the later measurements show a permanent deformation or not. (This last question might be detected by fitting two lines—the second one to a second complete set of points—and then inquiring whether or not these lines could have arisen from the same population.)

A more interesting experiment is the measurement of the thickness of a zinc coating on sheet steel. This can be found by a nondestructive repeatable test with a magnigage or destructively by dissolving off the coating, measuring the thickness before and after. If we cut up a sheet of metal into several specimens and perform these tests on each one, we are sampling from some kind of a population of coating thicknesses and the picture is further obscured by the errors of measurement. Perhaps Figure 2 is a reasonable model here, but with no control over what values of x we will obtain, that variation being due to the fluctuations in the thickness of the coating from specimen to specimen. The further complication that there can be several magnigage (y) readings for one stripping (x) measurement means that no unique (x, y) point can be found, rather the mean of the y values has to be paired with the one x value, and the individual y readings used to estimate part of the variation within the finest subdivision, that imperfectly known bivariate error distribution. This is an admittedly messy model.

A Berksonian model is found in the calibration of a refractometer by weighing out several preassigned amounts, x_i, of sugar in a balance, dissolving these in equal volumes of water and measuring the refractive index, y_i. Even if we weigh out several "one gram" samples with error, we have the classical model with error in y only, as we clearly cannot estimate errors in x if we consistently record x *equals one* in our book. The nice part about all this is that for determining the slope of the line connecting y and x, this omission is unimportant, and the line obtained is unbiased. Note that in using this calibration for predictive purposes y would normally be measured, and x estimated from the curve. The pertinent questions include confidence

[2] TUKEY, J. W., "Components in Regression," *Biometrics*, **7**, 33–69 (1951).

limits for the line as well as an investigation of the errors attributable to the volumes of water used and perhaps even the operator who carried out the calibrations.

Another Berksonian experiment might test a coefficient of expansion by heating a standard length metal bar until several standard elongations, y_i, were observed (with unrecorded error) and the temperatures, x_i, measured. Here the calibration is apt to be used in either direction, and we will have to worry about how far in error we might reasonably be if we used this curve either way.

If we measure the disturbances in radio transmission caused by sunspot activity, we have an example of an experimental law (linear, we hope) in which we cannot control the variables. Here not only are the variables random (the number of sunspots and the coefficient of transmissivity) but they are both measured with errors which are themselves different random variables. Clearly the analysis of this type of data might require different techniques from the models already mentioned.

Finally, let us return to several sheets of zinc-coated steel with different nominal thicknesses, u_i. Again magnigage and stripping would yield estimates of the thickness, and thus we would obtain several clusters of values (I can scarcely say points). If we plotted the mean magnigage reading versus the stripping value, we then get a point for each specimen, and a cluster of points for each nominal thickness. This is a quite complicated model, but certainly no worse than occurs in many other experimental situations.

Some models are easy to analyze; some are very difficult. It behooves the experimenter to ponder carefully the models he believes to correspond to his reality, then to design his experiment so as to take advantage of the easier analyses. Rarely can the statistician, from the data alone, decide what model is appropriate. To reveal the model in this way requires impracticable amounts of data. But though small numbers of points cannot prescribe their own model, nevertheless a properly chosen model will extract more meaningful information from those few data than would an improper one. Thus it is necessary for the man who knows the physical system, the experimenter, to think carefully about the model from the theoretical viewpoint, as well as in the light of his experience, *before he undertakes any measurements.* If the experimenter will force himself to decide on the appropriate model before taking the data, he will avoid some of those embarrassing situations where, at the conclusion of the experiment, the information sought is not present in the numbers recorded.

In conclusion, I should like to reemphasize a point which will need to be repeated many times: the line of best fit is, by itself, of very little use in prediction if we do not know *how good* that best really is. Without such knowledge almost any line will do. The eyefitted line used regularly by the engineer is not bad and need not be apologized for—*as a line.* It is only because this technique of fitting tends to throw away much useful information that we decry it. I reiterate: it is not the Line of Best Fit we want; on the contrary, the Lines of Worst Plausible Fit are needed for prediction, those lines which bound the region in which the unknown correct line might reasonably be found. With these we obtain a picture showing our degree of confidence, here we have a quantitative measure showing our degree of uncertainty, our safety factor if you will. The wider the plausible region, the less assurance we have that our use of the fitted line will give reliable results. The narrower the region, the more confident we feel. This is one of the purposes of the statistical theory, to make quantitative our confidence and hence prevent undue optimism about our knowledge in the face of a variable universe. The other main purpose is to examine the fluctuations called errors, and by examining them to improve our knowledge about the way experimental systems operate. In the examination of those errors, the line is an obscuring gross fluctuation to be removed and removed efficiently. For this purpose statistical techniques, frequently using sums of squared residuals, have been evolved and are very important. Their purpose is not so much to fit a best line as to exorcise it, thus uncovering the finer structure of the data and revealing the subtler information which is often hidden inside, unknown even to the experimenter.

When the experimental men understand these two purposes in statistically analyzing straight line data, the cult of Least Squares will be dead. If the experimenter can be persuaded to ask, "What errors probably enter into my experimental system, and how may they be described in a simple algebraic model?" then the major battle will be won. The useful techniques of statistics will then be applied by the man who is best suited to apply them, by the experimenter. The information extracted will repay the effort well, and the thought required by the analysis will lead to improved experimental designs. Gauss will no longer be honored as a Prophet, but rather as a scientist.

ERRATUM

On page 49 of the January issue is a review of "Vinyl and Related Polymers" by Schildknecht. The title is erroneously listed as "Vinyl and Related Plastics." We apologize to the author and publisher.

LECTURE DEMONSTRATIONS OF INCENDIARIES

MARVIN ANTELMAN
Yeshiva University, New York, New York

HERE are a few incendiary reactions to enliven lectures and to stimulate student interest.

Spontaneous Combustion. Mix 75 ml. of turpentine with 75 ml. of Japan drier in a 250-ml. beaker, and to this add rags or cotton waste. Allow some time for the chemicals to soak. Using forceps carefully remove the mass and place it in an evaporating dish. Measure the temperature at intervals. The temperature rises rapidly and will reach the kindling temperature in about 20 or 30 minutes. On no account should the treated rags or cotton wastes be touched with the hands. Unused material should be disposed in metal containers for obvious reasons.

Combinations of Chemical Elements. The action of sulfur with metals is incendiary, *e. g.*, ignition of zinc-sulfur mixtures. The final product is sometimes dissolved in dilute HCl to show students that H_2S is evolved when sulfides are treated with acids. Equally spectacular is the substitution of aluminum for zinc. The sulfide of aluminum differs from its zinc counterpart inasmuch that it is soluble in water, reacting with it to liberate H_2S. White phosphorus placed on iodine crystals takes fire spontaneously. Pea-sized bits of red or white phosphorus thrown into an evaporating dish containing a small quantity of bromine produce different effects, throwing up yellow flames as they touch the surface. Commonly used combinations of this type are the chlorine and oxygen combustions of metals and nonmetals.

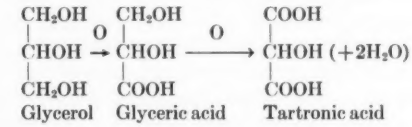
Reactions with Potassium Chlorate. The oxidizing effect of potassium chlorate is well illustrated by the so-called "Chemical Hotfoot." A 6 by $^3/_4$-inch test tube is placed securely in the jaws of a buret clamp from which the rubber sleeves have been removed. Fill up to $^3/_4$ inch from the rounded bottom portion of the test tube with $KClO_3$. Heat the tube until the chlorate forms a transparent melt. The clamp should now be screwed to a ringstand. Drop a wooden splint into the melt. A tremendous upshoot of flame and smoke takes place. After the reaction subsides add another splint, and the reaction is revived. As many as ten splints may be added to this small amount of material, producing heat and light each time. The number of splints added at a time may be varied. Sulfur-chlorate mixtures explode when hit with a hammer as do phosphorus-chlorate mixtures. It is unsafe to add sulfur or phosphorus to molten $KClO_3$.

Chlorine peroxide is a very unstable substance formed by the action of concentrated nitric or sulfuric acid on chlorates. Add a drop of concentrated H_2SO_4 to a little $KClO_3$ in an evaporating dish. The reaction is violent and spontaneous and may be accelerated by adding carbon, preferably in the form of sugar. There may be some spattering of acid if larger quantities are used. In the reaction chloric acid is liberated, which decomposes into chlorine peroxide as follows:

$$3HClO_3 \rightarrow HClO_4 + 2ClO_2 + H_2O$$

Although magnesium burns brightly in air and more brightly when in contact with oxidizing agents or oxygen, an almost explosive reaction occurs if magnesium is ignited with $KClO_3$ by the means of electricity. To illustrate this, powdered Mg and $KClO_3$ are mixed (do not grind) in portions of one part by weight Mg to 1.7 of $KClO_3$. Place the mixture in a crucible containing a resistance wire connected in circuit to a suitable current source controlled by a switch. It is a good idea to keep at a safe distance, extending the circuit as far as possible. When the switch is released the reaction will proceed instantly.

Glycerol-Permanganate Reaction. If potassium permanganate and glycerol are mixed to a thick paste, smoke is given off and combustion finally takes place. With 0.2 ml. glycerol mixed with one gram of permanganate fire occurs within 20 seconds. This reaction illustrates the oxidizing action of potassium permanganate according to the following equation:



Water Added to Mixtures. Mix iodine with equal amounts of Al, Mg, or Zn in three separate piles, and add a drop of water to each. Ignition occurs, and violet iodine vapors are given off. The heats of formation for magnesium, aluminum, and zinc iodides are 84,800, 70,300, and 49,231 calories, respectively. The place of these metals in the electromotive series is easily noticed by the vigor with which the combinations take place.

Water will also ignite a mixture of 500 mg. of zinc dust, 500 mg. of ammonium nitrate, and 200 mg. of ammonium chloride. Larger amounts can be used in the same proportions if desired. The reaction proceeds instantly after a drop or two of water is added. The mass smokes and then burns. Ammonia odors may be detected in the smoke.

Hydrogen Peroxide with MnO_2. Place a little MnO_2 in an evaporating dish, and allow a few drops of 30 per

cent hydrogen peroxide to fall upon the powder from a height of six inches or more. As each drop makes contact with the MnO_2, the H_2O_2 decomposes with a small explosion, releasing oxygen, and vaporizing the water present in dilute H_2O_2 as a cloud of steam. Exercise caution when handling 30 per cent H_2O_2 as it attacks the skin like concentrated acids.

Zinc and Sodium Hydroxide. Small quantities of zinc added to a dilute sodium hydroxide solution release hydrogen; but if the proportions of the reactants are varied considerably, totally different effects will result. Dissolve 10 g. of NaOH (flakes or pellets) in 95 ml. of water. Add powdered zinc until a thick paste forms. Squeeze the paste between sheets of absorbent paper to remove excess moisture. Spread the paste on an asbestos board (or other fireproof nonconducting surface) in order to form a layer $^1/_4$ inch thick. Combustion takes place at the end of two minutes. Three chemical reactions are involved here. (*a*) Zinc reacts with NaOH, (*b*) zinc is oxidized to its oxide by atmospheric oxygen, and (*c*) zinc oxide reacts with sodium hydroxide when moist. These three reactions accumulate sufficient heat to lead to combustion.

Volcanic Effects. Ammonium dichromate will decompose into green chromic oxide (Cr_2O_3) known commercially as "chrome green." If a quantity of the dichromate crystals are placed in the form of a mound and ignited, a volcanic effect is observed. Green bits of Cr_2O_3 and sparks are thrown into the air. At the end of the reaction, the green oxide, which is lighter than the dichromate, is left in a pile much larger than the original. Decomposition of $(NH_4)_2Cr_2O_7$ is expressed by the following equation:

$$(NH_4)_2Cr_2O_7 \rightarrow N_2 + 4H_2O + Cr_2O_3$$

Goldschmidt Reactions. Chromium is manufactured by the reduction of Cr_2O_3 using Al as the reducing agent in the Goldschmidt process. This process may be simulated on a smaller scale. Fill a crucible $^2/_3$ full of a mixture of powdered Al and Cr_2O_3. Place the crucible in a dish or pail of sand. On top of the Al and Cr_2O_3 mixture place a mixture of powdered magnesium and sodium peroxide. Now insert a piece of magnesium in the center and ignite it. Keep at least six feet away after lighting the magnesium. The ribbon burns downward igniting the peroxide mixture. This in turn ignites the aluminum, causing it to burn with almost explosive violence. Due to the nature of this reaction the crucible is liable to break. Metallic chromium is formed. In the thermite reaction oxides of iron are substituted for chromic oxide.

Nitrogen Halides. The chloride and iodide of nitrogen are interesting substances. Dulong, who discovered the chloride in 1811, lost one eye and three fingers in the process. Both substances are very unstable, being subject to explosion at the slightest touch. They can be prepared and safely exploded under proper condition. Nitrogen chloride (NCl_3) is a yellow oil. The iodide (NI_3) is in reality a black solid. A red-brown iodoazoimide ($2NH_3 \cdot NI_3$), commonly called nitrogen iodide, is very convenient to prepare and exhibits the same explosive properties.

To prepare NCl_3, fill a one-liter flask with chlorine gas (use a long necked flask if possible) and immerse the neck in a basin containing a saturated solution of ammonium chloride. Place a lead evaporating dish below the mouth of the flask. As the chlorine is adsorbed the solution will rise into the flask. When the flask is about three quarters full, NCl_3 will form on the sides and fall into the dish. By transferring the dish from the solution with clean tongs the chloride may be exploded by tickling it with a feather attached to a long glass rod. The chloride may be prepared by other methods, one of which is the electrolysis of a concentrated solution of NH_4Cl, the chloride being liberated at the anode. Chlorine reacts with NH_4Cl as follows:

$$3Cl_2 + NH_4Cl \rightarrow NCl_3 + 4HCl$$

When iodine crystals are dissolved in a solution of potassium iodide and the solution added to concentrated NH_4OH, a precipitate forms. This red-brown precipitate is a nitrogen azoimide. It will not explode when wet but when dried it explodes when tickled with a feather. The formation and explosion of the substance are expressed by the following equations:

$$6NH_3 + 3I_2 \rightarrow 2NH_3 \cdot NI_3 + 3NH_4I$$
$$2[2NH_3 \cdot NI_3] \rightarrow 3I_2 + 4NH_3 + N_2$$

Charcoal Burning with Absorbed Gases. One can strikingly demonstrate the ability of charcoal to absorb gases in the following manner. Fill a gas-collecting bottle with H_2S and two others with O_2. Fill two deflagrating spoons with charcoal, and place one in the bottle containing H_2S. Simultaneously ignite the contents of both spoons, and immerse them in the two bottles of oxygen. It can readily be seen that the charcoal containing the H_2S burns the most brightly.

Pyrophoric Substances. Certain substances are known as pyrophoric compounds or mixtures. If upon being heated in a test tube and their contents thrown out after heating, they catch fire. Some organic lead compounds have this tendency. To illustrate this, try using lead oxalate, citrate, or tartrate. Lead chromate and sulfur, alum and brown sugar, potassium sulfate and carbon, alum and flour, are all pyrophoric mixtures. The term pyrophoric is also applied to alloys such as misch metal and Auer metal, which emit sparks when scraped or struck.

Substances Producing Voluminous Ash. Some substances when heated leave a voluminous residue behind. Mercuric thiocyanate, sold as a novelty, or a mixture of potassium dichromate, nitrate, and sugar give this effect. For lecture purposes *p*-nitroacetanilide gives much better results. Place some of this yellow powder in an evaporating dish and moisten it with concentrated H_2SO_4 until a thick paste is formed. When this is heated on a ringstand over a Bunsen Burner, such a large quantity of ash is produced that it will climb out of the dish onto the base of the ringstand and creep along the lecture table. The circumference of this ash is equal to that of the evaporating dish.

NON-STABLE STATES

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THERE are many states which are not in true thermodynamic equilibrium either within themselves or with their surroundings. Those which undergo perceptible continuous physical or chemical changes have been readily recognized and classified as unstable states of varying degrees of instability. There are, however, many cases in which the particular state or phase is only *apparently* in equilibrium either internally or with its surroundings. In this field precise classification is difficult and, consequently, the available terms have frequently been incorrectly used. This is exemplified by the incorrect use of the term "metastable states." This term was introduced by Ostwald in 1897 (*1*) to cover such cases as supersaturated solutions and supercooled liquids which could remain for prolonged periods in such a state in the absence of any nuclei of their stable phases. Following this, Findlay (*2*) adopted the definition: "A system which in itself is stable, and which becomes unstable only on contact with a particular phase is said to be metastable." He also agrees with Othmer (*3*) that a very slow velocity of transformation of a given system cannot be used as a criterion for the system being truly metastable; the only criterion is that the addition of a nucleus of the "stable phase," or its spontaneous formation, should cause the change from the metastable into the unstable state. The two statements represent, however, different definitions since a "particular phase" and a "stable phase" can be different. Thus, in the case of supersaturated solutions, addition of the stable phase or of isomorphous substances can cause precipitation. However, the use of the word "particular" in the definition of metastable states, can lead to incorrect classification when it is used to refer to a nucleus which in itself is not the stable phase, but which nevertheless causes the metastable state to become unstable and so be transformed into the stable phase. Such use would lead to classifying as metastable certain states which are in apparent equilibrium, but where reaction can take place on the addition of specific catalysts. Thus, Taylor (*4*) considers a mixture of O_2 and H_2 at room temperature to be in a state of partial or metastable equilibrium. However, the introduction of some H_2O vapor or a drop of water into the mixture does not cause any combination, though the introduction of some platinum black results in reaction. If Taylor's use of the term metastable equilibrium is accepted then the definition has been extended to such an extent that it now implies that a metastable state is one which is in apparent or pseudo-equilibrium with the surroundings or within itself until energy of activation requirements have been met. If this be so, then the degree of metastability depends only on the magnitude of the energy of activation. Even finely divided solids would then have to be classified as metastable, since, in order to be able to pass readily into the stable state possessing minimum surface area (and therefore minimum surface free energy), they would have to pass into the liquid—or vapor—state first, thus requiring a certain energy of activation. On the other hand, acceptance of such a definition would appear to exclude cases where apparently no energy of activation is required, but where removal of the available free energy of the reaction is the primary condition for reaction to take place. For example, atomic hydrogen reacts according to the equation

$$H + H \rightleftharpoons H_2^*$$

where H_2^* is a "quasi-molecule" complex of the hydrogen molecule so "supercharged" with the energy of of the reaction that it redissociates into the two hydrogen atoms. The process

$$H + H \rightarrow H_2$$

cannot take place, therefore, unless a three-body collision occurs in which the third body removes the energy of the reaction (*5*). The "particular phase" in this case is then the third body, and the molecular hydrogen in the excited state may be termed metastable. Similar remarks apply to the combination of other free atoms such as those of nitrogen, chlorine, or bromine. Indeed, Kneser (*6*) expressed the view that the properties of activated nitrogen are to be attributed to the presence of the ordinary N atoms along with metastable molecules.

Other authors have extended the meaning of the term "metastable states" still further. Thus, Hackh's "Chemical Dictionary" (*7*) defines a metastable state as "an unstable condition which changes readily either to a more stable or a less stable condition," a metastable electron as "an electron moving in an excited orbit," and a metastable phase as "the existence of a substance as a solid, liquid, or vapor under conditions in which it is normally unstable in that state." This set of definitions has clearly extended the meaning of "metastable" to such an extent that it is synonymous with the term unstable. This, of course, is an unsatisfactory state of affairs.

Some investigators have adopted the term "metas-

table atoms" for those whose electrons are in excited states and which, for some reason, remain for some time in that state. This extension of the term metastable state seems to have been widely accepted (*8*), probably for want of more suitable terms, despite the fact that excited atoms of this kind may proceed to emit radiation and therefore pass into a lower free energy state in absence of any specific nuclei. The main reason for placing these atoms in the category of metastable states would appear to be the consideration that the time interval between the attainment of this state and the eventual transformation into the lowest energy state is longer than usual.

Several workers, including Findlay (*9*), use the term "metastable metals" for metals whose properties have altered on being subjected to mechanical treatment. Yet it is not certain that these "metastable" metallic states would tend to break down *only* on the addition of—or self-formation of—the required stable state or some particular nuclei. The inclusion of these metallic states in the category of metastable ones may be considered correct, if we define a metastable state simply as an unstable one which proceeds *very* slowly to an equilibrium state. But, acceptance of this latter definition would result in the original significance of the term *metastable states* being completely lost, since no nuclei of the stable state are required, and would still leave unclassified the cases of atomic hydrogen, "metastable atoms" (since if radiation is emitted, it occurs instantaneously), and other cases.

This unsatisfactory state of classification has resulted from loose use of the terms "metastable" and "unstable" and from the lack of suitable definitions of the various states which are not in true thermodynamic equilibrium either internally or externally. A possible contributory factor has been the tendency to model the terminology of the various energy states on physical models where the usual terms employed are: stable, unstable, metastable, and, sometimes, neutral, despite the care required in such analogies. Now, in all the previous cases the free energy content of each is above the minimum possible under the experimental conditions given. At present, such systems are said to be thermodynamically unstable. But the significance generally attached to the word unstable is that an unstable system is in the process of losing its excess free energy and is therefore undergoing transformation into the thermodynamically stable state. This leads to confusion, because many systems which are not in equilibrium do not undergo such transformation, and hence they have been classified as metastable. Any classification of non-equilibrium states would be made easier if the word unstable were reserved for cases such that can be shown to be undergoing, however slowly, transformation into the stable state. A new term would then be required to cover all non-equilibrium states; and the various cases could then be placed in subdivisions. To this purpose the following definitions are suggested.

THERMODYNAMIC NON-STABILITY

A thermodynamically non-stable state is defined as a state which is not in true thermodynamic equilibrium either within itself or with its surroundings. Thermodynamically non-stable states are therefore those in which $(\Delta F)_{p,t} < 0$, or $(\Delta S)_{e,v} > 0$. Such states may be divided into the following four categories:

UNSTABLE STATES. An unstable state is a non-stable state which is undergoing continuous transformation into states possessing lower free energy contents (or greater entropy contents). Examples are:

Instability at Constant Temperature. (1) Any substance or substances undergoing reaction at a noticeable rate at constant temperature and pressure.

(2) Isothermal expansion of a gas.

(3) Vector distribution: *e. g.*, a gas "at constant temperature" in which the average kinetic energy of the molecules obeys *numerically* the Maxwell-Boltzmann distribution, but where the vector distribution is unequal (*i. e.*, where the equipartition principle is not obeyed). Thus, in the case of a molecular beam, the molecules are at "constant temperature," but their directional movement is restricted. In a closed space they would tend to assume the Maxwell-Boltzmann distribution in all directions.

(4) Diffusion of solutes from concentrated solutions into less concentrated solutions.

Temperature Differences: e. g., a hot body in contact with the surroundings which are at a lower temperature.

PSEUDO-STABLE STATES. A pseudo-stable state is a non-stable state which *appears* to be in equilibrium within itself or with its surroundings, but which nevertheless is slowly, or very slowly, changing continuously into the lower free energy states possible. Examples of this are:

(1) Lyophobic colloids: These tend to coagulate very slowly.

(2) Finely divided solids: These tend to form large crystals having minimum surface area.

(3) A mixture of H_2 and O_2 at room temperature: The formation of water, in the absence of a catalyst, is extremely slow.

(4) A magnetized bar of steel: It tends to be demagnetized very slowly.

QUASI-STABLE STATES. A quasi-stable state is a non-stable state in which "reaction" takes place readily but which is readily reversed, because the products retain in full the free energy of the reaction. This reversible reaction—or alternation of the system between two states which have the same free energy content—continues until the removal of the free energy change available takes place on the introduction of some suitable material. Examples are "quasi-molecules" of hydrogen, chlorine, bromine, or nitrogen.

METASTABLE STATES. A metastable state is a non-stable state in which no transformation into the stable

state can take place until the self-formation—or the introduction—of a nucleus of the stable phase takes place.

This definition excludes nuclei which cannot cause the internal formation of a nucleus of the stable phase. It also implies that the formation of a nucleus of the stable phase is the primary requirement of the change from the metastable into the stable state to take place. This definition, therefore, includes any agent that can cause the formation of a nucleus of the stable phase, *e. g.*, isomorphous substances, and excludes quasi-stable and pseudo-stable states which do not require a nucleus of the stable state for reaction to proceed.

Examples are supersaturated solutions or supercooled liquids or solids, all of which require the presence of a nucleus of the stable phase for transformation into the stable phase to proceed.

ENERGETIC NON-STABILITY

The above classification was based solely on thermodynamic grounds. Consequently, atomic non-stability and mechanical non-stability could not be included. Cases belonging to the latter categories, *e. g.*, atoms, in electronically excited states which are not radiating any energy, and a falling body, which is undergoing elastic collisions with the ground, can be classified in *energetically* non-stable states. There are, of course, several types of mechanically non-stable states; they can be placed in subdivisions parallel to those proposed for thermodynamically non-stable states. But the case of excited atoms is quite distinct, and for this reason a new term is proposed.

MESO-STABLE STATES. An atom is said to be in a meso-stable state when, despite being in an electronically excited state, it does not emit radiation and fall into a lower free energy state. A meso-stable state is then an *energetically* non-stable state in which no continuous change takes place. (In this latter respect it is somewhat similar to a metastable state.)

Other types of classifications and subdivisions have been considered by the writer; but they do not seem as clear or as advantageous as the proposed categories, and they are open to strong criticism. On the whole the definitions and subdivisions adopted in this communication are open to less criticism than other classifications and it is therefore suggested that they should be adopted for general use.

This is not, as it may seem, an academic point of classification. There are important implications arising from it in the theory of precipitations and other reactions which may be discussed in later publications.

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TRADE SECRETS

THE following, reprinted from *The Chemical Age*, December 30, 1950, would seem to be good reading for a young chemist about to go out into industry.

The return to normal competitive conditions makes it more than ever necessary that individual processes and methods of work should not be passed on to rival organizations. Responsible employees are naturally in a position to prejudice a business by giving its secrets to rivals and it is helpful for employers to know the way in which the law safeguards their position.

The law appreciates that mutual confidence between employer and employee is essential and a term is therefore implied in all contracts of service, whether written or oral, that the employee will act with good faith. This duty arises independently of any express terms in the contract of service, but express terms may of course, widen the duty, and their effect will therefore require subsequent examination.

The implied term forbids breaches of faith by the employee both during a particular employment and after it has ended. The master has several available remedies against an employee who breaks faith. Dismissal without notice may be given and the employer can obtain an injunction against the employee and any collaborator, restraining them from profiting by the employee's breach of duty. The employer may also be entitled to damages.

As well as preventing the passing on of confidential matters, the implied duty obliges employees to report the misconduct of fellow-employees, regardless of the offender's status. By virtue of their position, executive and over-seeing staff have to report all acts of misconduct, the duty varying with the nature of their employment and their duties. The average employee, however, need usually only report acts which are criminal, fraudulent, or clearly wrong.

DIFFICULT TO DEFINE

The implied term also forbids the communication of "confidential information." This is rather difficult to define with precision. One concern may consider its costing system highly confidential, while another would attach little importance to the system, but much to the way in which a particular costing has been built up. The application of the term largely depends on the facts of each case, and decisions of the Courts provide a useful guide.

In one case, an employer was granted an injunction restraining his former employee from using a copy of his order book for the purpose of soliciting orders for a rival firm and was also awarded damages. Again, where the employee of a firm of engine manufacturers secretly made notes of the engine dimensions, the employers were granted an injunction restraining him from communicating this information to other persons.

RIGHT TO GOOD FAITH

When the implied duty is being considered in relation to borderline cases, where it is not clear whether confidential information is involved, the general policy of the law that employees should be free to acquire as much skill and knowledge as possible in their trade or calling, must be kept in mind. However, the law recognizes that employers have a right to good faith from their employees, and in three cases, information, no matter of what type, is always confidential.

First of these is when it is an express term of the contract that it should be so. Secondly, where the nature of the employment is such that a particular matter would obviously be defined as confidential. Thirdly, where information is surreptitiously acquired by the employee. Outside these three cases, it seems that the employee may make use of such information as he might reasonably be expected to remember. Thus an employee may canvass his former employer's customers so long as he does not make use of information acquired other than by the habit of dealing with those customers. In other words, that he does not use lists compiled during his employment.

This implied duty to keep good faith is generally an adequate safeguard to the employer, but definite terms may be necessary in the case of technical experts and managerial grades. A formal contract of service with such employees is advisable and a term putting a restraint on their future activities when their employment ends, can be included. For such restrictions to be legally enforceable however, the employer must be able to show that they do no more than is necessary to give reasonable protection to his proprietary interests and are not against the public interest. He must show that the area or space restriction and the period for which it is to last, are reasonable.

Under this rule, a covenant by the manager of a concern using a secret process to manufacture glass bottles, that he would not serve a competing concern for five years, was upheld as being reasonable. On the other hand, a covenant by a canvasser in Islington, prohibiting him from entering similar employment within 25 miles of London, was held to be unreasonable and invalid.

Restrictive covenants give employers an added measure of protection, but the law concerning them is complex. Therefore, unless it is merely intended to prohibit an employee from entering the service of named competitors, it is advisable to obtain professional advice in drafting the covenant.

A VACUUM DRYING APPARATUS FOR SMALL SAMPLES

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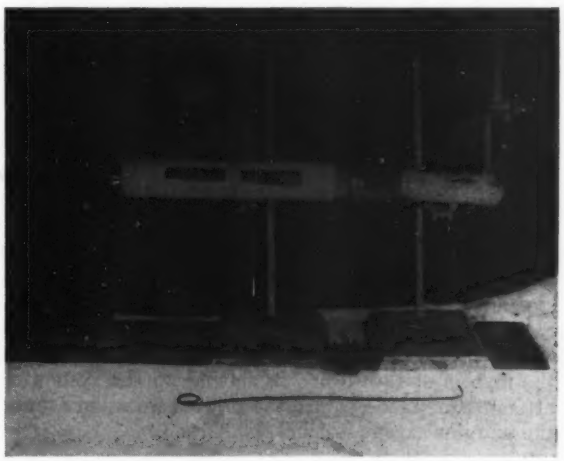
THE Fisher Pistol has been the standard apparatus for vacuum drying of small samples. This piece of apparatus is rather difficult to construct, and the solvents which are required are often lacking or contaminated when needed.

A substitute for the traditional apparatus is an electrically heated Pyrex tube. This piece of apparatus may be constructed by a glass blower of moderate skill; it requires no solvents and may be permitted to run safely overnight.

The illustration shows the equipment which has been used for a year. It has been run for periods up to 24 hours, and at temperatures up to 100°C. Its construction requires materials which are readily available to any laboratory.



CONSTRUCTION

Materials: Pyrex tube 34 mm. O.D.; a standard-taper 35/40 joint; 30 feet of nichrome wire, B & S No. 22; variable resistance of 200–250 ohms; a Pyrex three-way stopcock; sheet asbestos; ring stands; clamps; wire gauze; and a board for a base.

The asbestos sheet is first wrapped about the Pyrex tube and secured by three copper wires. The nichrome wire is then wrapped about the tube, 10 turns per inch, and the ends are fastened with copper wire. The second layer of damp asbestos is then wrapped over and secured as was the first. The asbestos-wrapped tube is then dried in an oven at 110° for about 24 hours.

The wire gauze is bent into the shape shown in the picture to serve as a vial carrier. This places the vial in the center of the heated tube.

A PUNCHED-CARD SYSTEM FOR GRADUATE RESEARCH

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VERY SOON in the career of a graduate student the background information, data, and information about experimental techniques relevant to the graduate research problem accumulate to such an extent that informal notes are no longer sufficient to keep the material readily available. Faced with this problem, the student usually attempts some scheme of classification to provide an orderly way of filing and reclaiming the information. Almost immediately he runs into the difficulty of classifying an abstract of an article. To file the information properly requires either an arbitrary selection of the most important piece of information and filing the card under that heading, or making separate cards for each item of information and filing these separately. The first method tends to lose information and the second method tends to make the file voluminous and unwieldy.

The means of avoiding the loss of information and of holding down the size of the files is found in the use of hand-sorted, marginally punched cards. These punched cards make possible multiple classification of an abstract or even an individual item of information. The problem is then reduced to deciding upon a coding system which will permit "filing for finding." In the material that follows the system set up by the author for his graduate research is outlined.

The card used in this system (see the figure) is type KD585B, made by the McBee Keysort Company of Athens, Ohio. Its dimensions are 5 × 8 inches, with approximately 4 × 7 inches on each of the two sides available for recording information. There are two rows of holes punched around the margin giving a total of 182 holes for coding.

On the card shown in the figure there is a copy of an abstract concerning the deposition of cobalt-tungsten alloys. At the bottom of the card there is a notation in pencil of the code for that particular card. The code letters and numbers refer to particular holes around the margin of the card. In setting up the system no attempt was made to use the printed notation around the margins except to label the holes.

THE AUTHOR CODE

It was decided to use the first letter of the senior author's last name in the author code. There being 26 letters in the alphabet a numerical code was selected which would give 26 items with as small a number of holes utilized as possible. The numerical code is

1	3	9	outer row
2	6	18	inner row

This numerical code can be generated for as many pairs of holes as is desired by the relation

3^n	outer row
2×3^n	inner row

The code has the following rules: (1) a hole punched either on the outer or inner row may not be punched a second time; and (2) to obtain a number, select the code numbers which are the largest factors in the sum equal to the number desired, with such exceptions as required by (1).

For example, to code *E*, punch holes for 2 and 3; to code *V*, punch the holes for 1, 3, and 18. On the card the holes along the upper margin at the left end marked *A*, *G*, and *A* are used for the author code. On this particular card, the senior author's last name begins with the letter *C*, therefore the hole for 3 is to be punched. The pencil notation G_0 refers to the hole for number 3, *i. e.*, *G* labels 3 on the outer row (subscript$_0$) and 6 on the inner row (subscript $_1$).

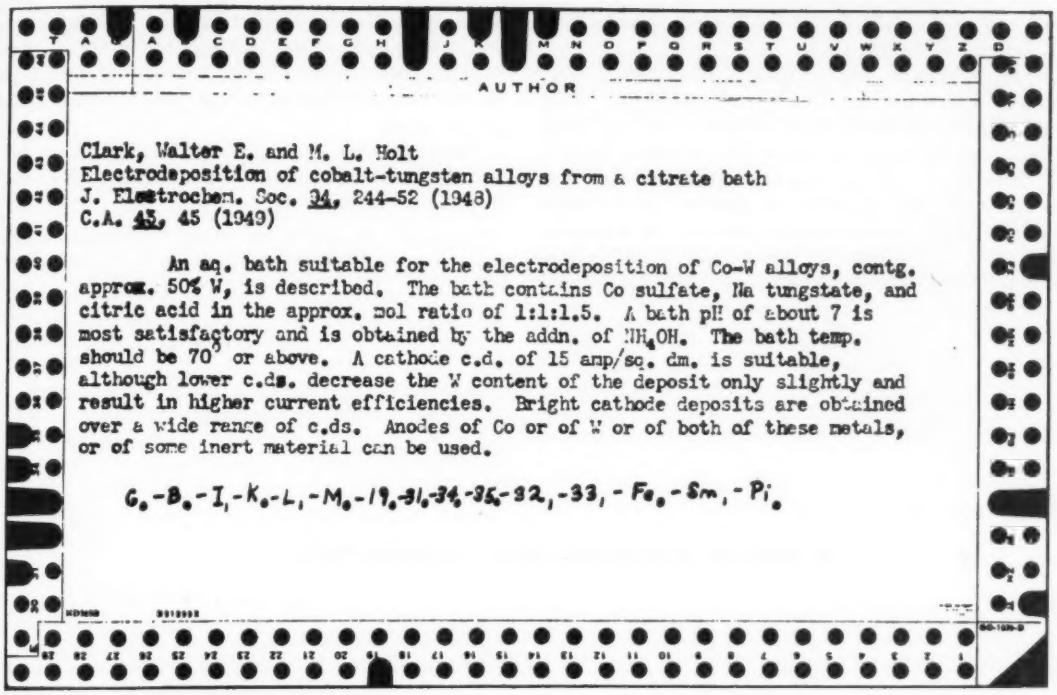
THE SOURCE CODE

The value of a source code may be questioned, but it was felt that it would be used enough in searching for information to warrant its addition to the card. To utilize the source code, separate 3-× 5-inch file cards are maintained, with the cards filed alphabetically according to source. This permits the assignment of a number to each source as it appears in building up a file. The same numerical code as was used for the author codes is used in coding the sources. The holes labeled *B* through *H* along the top margin of the card are reserved for the source code, permitting a total of over 2200 sources to be coded if necessary. At the present time over fifty separate sources have been included. On the card in the figure, the source is the *Journal of the Electrochemical Society*, which happens to be number 1 (noted on the card as B_0).

To distinguish between patents and all other types of sources, use is made of the hole labeled *T* in the upper left hand corner of the card. If the card is made from a patent, the hole is punched, otherwise it is left unpunched.

THE DATE CODE

The date code was included for the same general reason as the source code, *i. e.*, seeking information when only the date and some other fact is known about the particular card. In the date code the first outer hole, I_0, corresponds to 1800 and the first inner hole, I_1,


AUTHOR
Clark, Walter E. and M. L. Holt
Electrodeposition of cobalt-tungsten alloys from a citrate bath
J. Electrochem. Soc. 94, 244-52 (1948)
C.A. 43, 45 (1949)
An aq. bath suitable for the electrodeposition of Co-W alloys, contg. approx. 50% W, is described. The bath contains Co sulfate, Na tungstate, and citric acid in the approx. mol ratio of 1:1:1.5. A bath pH of about 7 is most satisfactory and is obtained by the addn. of NH4OH. The bath temp. should be 70° or above. A cathode c.d. of 15 amp/sq. dm. is suitable, although lower c.ds. decrease the W content of the deposit only slightly and result in higher current efficiencies. Bright cathode deposits are obtained over a wide range of c.ds. Anodes of Co or of W or of both of these metals, or of some inert material can be used.
G0-B0-I1-K0-L1-M0-19-31-34-35-32-33-Fe0-Sn1-Pi0

to 1900. The remaining holes in the group *J* through *N*, provide spaces for the ternary code used for the author and source codes. The date of the article in the *original* reference on the card is 1948 so the holes corresponding to 1900, 3, 18, and 27 (I_1, K_0, L_1, and M_0) are punched.

THE SUBJECT CODE

Before setting up the subject code, a relatively large number of references was collected, then an outline of the subject material desired was drawn up. It was found that some items could be grouped under one major heading while others had to stand alone. A direct code, *i. e.*, one-to-one correspondence between holes and topics, was chosen as being the least ambiguous way of coding a relatively large group of topics. There are at present 76 individual headings and 32 headings for individual materials, metals in this case.

In the reference on the example card specific plating practice as regards acidity, concentration, current density, electrode metal, and temperature is indicated. The code as set up has a major division for specific plating practice. The subtopics are:

Acidity	Geometry of the cell
Addition agents	Surface structure and metal of the electrode
Agitation	Temperature
Concentration of electrolyte	Type of electrolyte
Current density	Pretreatment
Current type	

The subject code numbers are assigned to the outer and inner rows of holes on the bottom and left hand side of the card, which are numbered from 1 to 46. The major division referring to specific plating practice is located from numbers 31 to 36, outer and inner holes. The topics listed in the left hand column above correspond to the outer row of holes and the ones listed in the right hand column to the inner row. The pencil notation on the card—31_0-34_0-35_0-32_1-33_1—thus refers to specific plating practice about acidity, concentration of electrolyte, current density, metal of electrodes, and temperature.

The code numbers for the metals are assigned to the outer and inner rows of holes along the right hand edge of the card labeled with the chemical symbols for some of the elements. The pencil notation on the card—Fe_0-Sn_1-Pi_0—refers to cobalt, tungsten, and alloys.

CODING AND PUNCHING

In coding a card the reference is typed on the card first, then the reference is checked against the subject code and pencil notation in code of the items present is made on the card. Finally, the holes are punched out. The punch used is the 5202 hand punch, a so-called deep punch, sold by the McBee Company.

In sorting for an author, a source, or a date, a sorting probe (a knitting needle works well) is pushed through the hole, in a stack of cards, which corresponds to the highest factor in the sum equal to the number searched for. All cards having that position punched then drop when the needle is lifted. The next highest factor is then sorted for and so on until the card, or a small group of cards remain.

To sort for a specific topic or metal, the probe is inserted in the hole corresponding to that topic or

metal and the needle lifted. All cards punched for that position will then drop.

The inner hole on some cards will be punched in the same position as the outer hole being sorted for. Both types of holes will cause cards to drop when the needle is lifted. In that case the outer hole is sorted for first, then those cards which drop are sorted for the inner hole. In that case all cards *not* punched on the inner hole will remain on the sorting probe. In that part of the card containing the subject code some cards will be punched both in the inner and outer holes (the direct punching rules permit this) so that the cards which dropped on the last sort made above, if a sort is being made in the subject region of the card, should be hand sorted, looking at the pencil notation for the proper label.

The book by Casey and Perry[1] supplies a great deal more information for the formulation of systems such as the one here discussed.

[1] CASEY, R. S., AND J. W. PERRY, "Punchard Cards, Their Applications to Science and Industry," Reinhold Publishing Corp., New York, 1951.

A USEFUL MELTING-POINT APPARATUS

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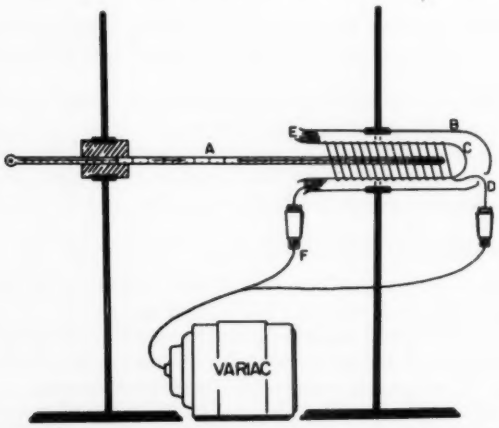
THE schematic diagram illustrated here describes a simple, rapid, economical, and accurate melting-point apparatus. Its advantages are that it requires no capillary melting point tube, no oil-bath medium, the temperature may be raised at any desired rate from 30° to 450°C., it may be cooled down within five minutes from 360°C., and it may be cleaned rapidly. The only piece of relatively expensive equipment the apparatus requires is a Variac; however, almost every laboratory has one. The remainder of the equipment consists of two test tubes, a few feet of nichrome wire, two battery clips and a foot of asbestos ribbon.



A. Thermometer mounted by a clamp on a separate ring stand
B. Outer test tube
C. Inner test tube wound with nichrome wire (*D*)
E. Asbestos ribbon to support tube
F. Clamps and electric cord to Variac

There can be several modifications of this apparatus, but here a simple, fast, and economical method for its preparation is described.

A 150- × 18-mm. test tube, wound as illustrated with 0.032-in. 20-gage, 0.63-ohm/ft. nichrome wire and moist asbestos ribbon, is forced into a 200- × 25-mm. test tube in which a hole has been blown at the bottom. The entire chamber is dried in an oven. When dry, the apparatus is assembled as shown in the figure.

To determine a melting point one places a few crystals in the center of the mercury bulb of the thermometer (*A*) with a spatula. The entire thermometer assembly is guided into the heating-chamber assembly until at the position illustrated in the figure. The dial on the Variac is set to about eight volts if the melting point range is below 150°C. If the melting point is above 150°C., the voltage is set proportionately higher. When the temperature approaches the melting range, one may turn the voltage down to about three, thus allowing the temperature to rise very slowly (about two degrees per minute). When the melting point is reached, the thermometer assembly is removed and the residue wiped off the bulb with a cloth moistened with alcohol or acetone. The apparatus is then ready for another determination.

A SIMPLE RECORDING POLAROGRAPH[1]

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THE problem in any recording polarograph is to apply a uniformly increasing potential to the electrolysis cell and to then record the current through the cell as a function of this potential. The following is the description of an apparatus that accomplishes this and which can be easily constructed in the average laboratory.

A U-tube of glass, inside diameter 12 mm., was constructed. The long arm measured 110 cm., the short arm 70 cm. The two arms were tapered down and joined to 7.5-mm. glass tubing. A three-way stopcock was located at the junction of the arms and the third lead, consisting of a short piece of glass tubing, formed a drain for the arms of the U-tube. Details of the apparatus are shown in Figure 1. The U-tube was mounted on a backboard. Within each of the arms a length of 28 B + S chromel resistance wire was strung, the terminals leading through glass seals. The length of wire in the short arm was 50 cm. while the wire length in the long arm (above the upper terminal in the short arm) was also 50 cm. The length of wire in the long arm which overlaps the wire in the short arm is immaterial.

This apparatus can be used to apply a uniformly increasing potential in the following manner. Mercury is placed in the lefthand arm reaching just to the top of the resistance wire, and in the right hand arm reaching just to the bottom of the resistance wire. Initially, the stopcock is set to keep the two columns of mercury separate. A source of potential is then connected to the top terminals of the two resistance wires; and the electrolysis cell circuit, consisting of the electrolysis cell and galvanometer with suitable shunt, is connected across the resistance wire in only the long arm. The stopcock is then opened and mercury is allowed to flow slowly into the short arm until the mercury levels in the two arms are equal. This requires eight to ten minutes. Then, by means of the three-way stopcock, the mercury is removed from the short arm and placed in the long arm for another run.

Initially, the potential applied across the electrolysis cell circuit will equal the potential drop across the column of mercury covering the resistance wire in the long arm of the tube. This will be small and it is dependent upon the magnitude of the potential applied. As the mercury drops, uncovering the resistance wire in the long arm and simultaneously covering the short arm resistance wire, an application of a uniformly increasing potential to the electrolysis cell circuit results. This potential will reach its maximum when the levels in the two arms are equal, and will be approximately equal to the potential applied across the terminals of the resistance wire. It should be noted also that the potential applied to the electrolysis cell circuit is a linear function of the level of the mercury in the long arm. The initial potential drop corresponding to the column of mercury can be readily determined in each case by a voltmeter. The final maximum potential across the electrolysis cell circuit will be less

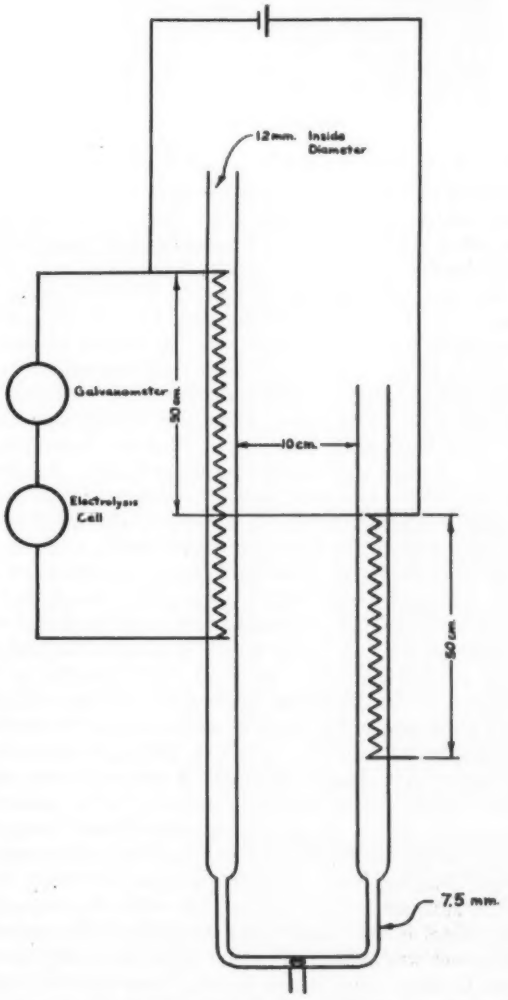

Figure 1. Sketch of Apparatus

[1] Presented before the Annual Meeting of the Virginia Academy of Science, May 17, 1952.

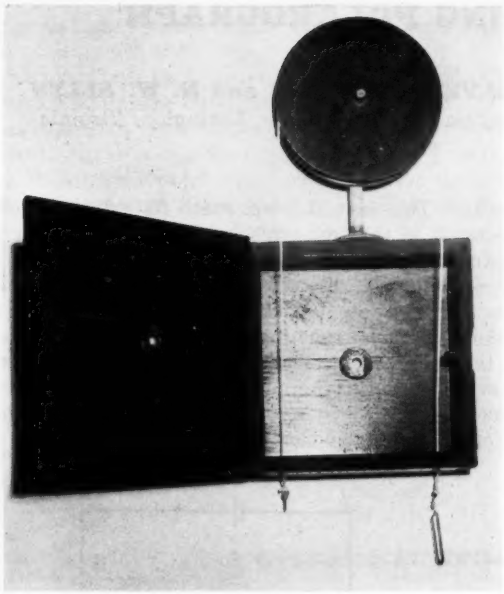

Figure 2. The Camera

than the potential applied across the terminals of the resistance wire by the potential drop that occurs along the entire column of mercury. The final maximum potential applied can be determined exactly by means of a voltmeter.

In order to record the current in the electrolysis cell circuit as a function of the potential applied, the following arrangement was employed. A camera consisting of a box 24-cm. square and 5-cm. deep was constructed. One side of the box was hinged and a fine slit was cut in this side. The edges of the box facing the hinged lid were faced with velvet so as to make the box light tight. A catch held the lid tight when closed. Mounted in this box was a solid grooved wheel of wood which could be rotated, the circumference of the wheel being equal to the distance through which the mercury drops (50 cm.) in the U-tube previously described. Cord was wrapped around the groove of the wheel, and its ends were brought through two small holes in the bottom of the box. The length of the cord outside the box must be greater than 50 cm. Details of the camera are shown in the photograph in Figure 2. In this photograph the wheel is shown outside of the box, resting on an iron arm that was used in mounting the camera. Attached to the ends of the cord there were two weights, the heavier consisting of a plummet (mercury sealed in glass) and the lighter, simply a small weight sufficient to keep the cord under tension when hanging free. The two weights, however, must differ sufficiently in weight so that when they hang free the wheel in the camera rotates easily. The mercury plummet was sufficiently small to fit inside the bore of the U-tube. The whole camera was painted black. The slit in the lid had projections on both sides as indicated in Figure 2. The inside projections just cleared the circular wheel by the thickness of photographic paper.

The galvanometer in the electrolysis circuit was of the light-reflecting wall type, and light from an ordinary galvanometer lamp was employed. A narrow slit of light was obtained by placing a piece of cardboard, with a narrow slit cut in it, just in front of the lamp bulb.

To record a polarogram, photographic paper is cut to correspond to the size of the wheel in the camera and it is mounted on the wheel. A thumb tack near the center prevents the paper from slipping during a run, and a piece of tape over the slit keeps the box light tight after removal from the dark room. The camera is mounted above the U-tube previously described, with the mercury plummet floating on the column of mercury in the long arm. The counterweight is allowed to hang free. The slit of light reflected from the galvanometer mirror is adjusted so it plays on the slit of the camera cover near the center of the photographic paper, and is perpendicular to the slit in the camera. With all adjustments made, the tape is removed from the slit about one minute before the start of a run in order to record zero position. To make a run, the stopcock is opened and the mercury allowed to flow slowly into the short arm. As the mercury drops in the long arm, the mercury plummet drops, rotating the wheel upon which the photographic paper is mounted. This results in a polar type of polarogram, in which the angle of rotation from the initial position is proportional to the applied potential and the distance from the center is a measure of the current. This is illustrated in Figure 3. At the completion of a run, the tape is replaced and the camera re-

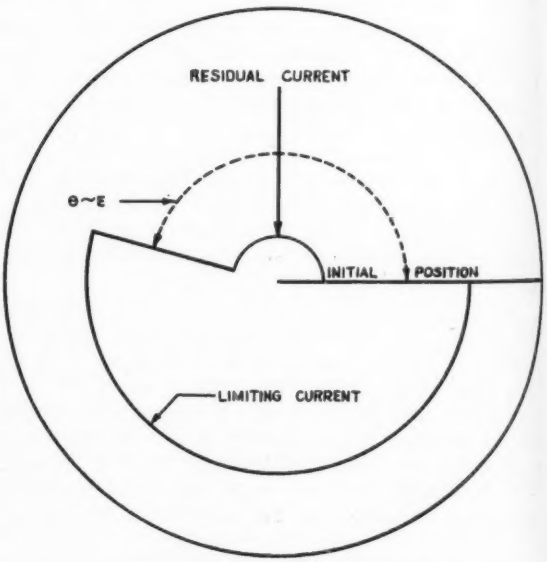

Figure 3. Typical Polar Polarogram

moved to the dark room for development of the polarogram. Runs were made in a lighted room with no noticeable fogging of the polarogram.

To determine the half-wave potential the angle, O_1, from the initial position to the midpoint of the break is measured. Then, if O_2 is the angle corresponding to the complete uncovering of the resistance wire in the long arm:

$$\frac{O_2}{O_1} = \frac{\text{Span e. m. f.}}{E\frac{1}{2}}$$

In exact work, the potential drop corresponding to the column of mercury covering the wire in the long arm should be added to the calculated half-wave potential. Any particular set of apparatus must be calibrated for the current. Then the distance between the circle corresponding to the residual current and the limiting current can be translated into terms of i and the concentration.

In our apparatus a galvanometer with a sensitivity of 0.01μ amp./mm. at 1 meter, and critical damping resistance of 10,000 ohms was employed. It was located at a distance of 50 cm. from the camera.

If a conventional rectangular type of polarogram is desired, this can be accomplished by a camera arrangement consisting of a rotating cylinder mounted in a light tight box of suitable dimensions. In this case, the axis of rotation would coincide with the axis of the cylinder. The whole apparatus can be made less bulky by coiling the resistance wires located in the U-tube, and gearing the camera so as to obtain a complete rotation.

FLUORESCENCE TITRATION

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De Ment Laboratories, Portland, Oregon

THE titration of opaque, highly colored, and similar solutions has always been a problem in analytical chemistry. Such a problem is often solved by the use of fluorescent indicators. Fluorescent indicators are substances which show definite changes in fluorescence with change in pH, provided an end point occurs. Fluorescent indicators may change from one fluorescent color to another, or the indicators may serve because of the appearance or disappearance of fluorescence at certain pH values.

Fluorescent indicators often have excellent and sharp end points. As such indicators are usually organic compounds such as dyestuffs and alkaloids, the converse approach permits in many instances the fluorochemical assay of the indicator itself. This can be especially valuable in medical work where, for example, quinine can be determined (*10*, *16*). Thus, the intensity of the fluorescence of quinine in dilute sulfuric acid at room temperature and pH 2 bears a linear relationship to the concentration (*5*). In fact, quinine is often used as a standard in the more elaborate fluorometric methods which involve instruments for this purpose.

Fluorescence titrations are best carried out in semi-darkness or in total darkness. A long-wave-length ultraviolet lamp, equipped with a filter, replaces the usual source of light. A few particles of insoluble fluorescent material are dropped onto the surface of the liquid in the buret, in order to follow the meniscus. Or, a few particles of fluorescein or quinine on the surfaces of basic and acidic solutions, respectively, can be used.

There are many fluorescent indicators available and the techniques can often be varied to fit the problem (*cf. 9*, *3*). The organic indicators are of most general value, whereas in special problems inorganic indicators are frequently helpful. Uranyl salts have been proposed and used from time to time, but the influence of various ions upon the fluorescence of uranyl solutions is usually too great to permit accurate work. Considerable study has been done in this direction on the halogen ions and others (*32*).

In place of methyl orange or methyl red in bromide-bromate titrations fluorescein has been found satisfactory, as its fluorescence is not so likely to be destroyed by free bromine (*17*).

If a small amount of freshly prepared 5-aminosalicylic acid solution is added to sodium hydroxide solution and this titrated under ultraviolet light, the fluorescence disappears as soon as all of the base has been neutralized. The results obtained agree with those for methyl orange (*21*).

Fluorescent indicators may exhibit shortcomings

common to other organic indicators. Certain fluorescent substances react with heavy metal ions, diminishing their effectiveness in titration. Nevertheless, this unique class of indicators is often profitably relied upon in specialized or unusual problems.

In fluorescence titrimetry the buret and other glassware, especially the receiving vessels, should be nonfluorescent, so that clear results are obtained. The glassware stock of the laboratory is first examined under ultraviolet light and those articles which do fluoresce are not employed.

APPLICATIONS

Many branches of chemistry benefit by adoption of fluorescent indicators and the ultraviolet lamp. The lamp itself is a valuable tool which can be applied to many other problems, aside from those of volumetric analysis (*cf. 3, 10, 9, 18, 29*). The opaque, turbid, or deeply colored liquids which are titrated under ultraviolet light should not exhibit an intense fluorescence of their own, as this interferes with the fluorescence of the titration. In some instances, the addition of a quencher, an agent which reduces or destroys the fluorescence, facilitates titration. The best quenchers are usually compounds with nitro groups or with several halogen atoms.

In particular, the food industries and the fermentation arts find fluorescence titration valuable. Examples include colored wines and other liquors, fruit juices, dark vinegars, and plant extracts. In pharmacy, colored tinctures and extracts yield to the method. In agriculture, soil extracts and like samples can be titrated.

Fluorescence titrations can be employed to advantage in microchemistry and ultramicrochemistry where exceedingly small amounts of liquid are handled, often with the assistance of the microscope. A fluorescent substance also helps in following a liquid in a capillary tube or in a microburet, and is useful when colors or color changes cannot be easily discriminated or are apt to be deceiving.

The determination of the ionization constants of complex organic compounds has been claimed feasible by methods similar to fluorescence titration (*29, 14*). The measurement of ionization constants for such substances as quinine sulfate, methoxyquinoline, and β-naphthol has been found to be in agreement with values obtained by other techniques.

The fact that isomers of various organic compounds vary in their fluorescent qualities with respect to pH provides a new and quick method of distinguishing such substances. The same is true of complicated organic molecules which vary slightly in composition or structure. In such methods fluorescence intensity is plotted against pH.

Thus, the α- and β-naphthylamines vary; also the many naphtholsulfonic acids, and the naphthionic acids (*9*). The various eosins, for example, show different fluorescent responses with changes in pH.

CATALOGUE OF INDICATORS

In the following list of some 60 fluorescent organic indicators a division is made according to the approximate pH range covered.

pH 0 to 2

Benzoflavine: yellow fluorescent at pH 0.3, changing to green at pH 1.7 (*19*).

3, 6-Dioxyphthalimide: blue fluorescent at pH 0, changing to green fluorescent at pH 2.4 (*19, 18*).

Eosin: colorless at pH 0 and green at pH 3.0 (*6, 18, 27*).

Eosin G: similar to above, having the same transition points, except that the fluorescence is yellowish at pH 3.0 (*12*).

Eosin YS: yellow colored at pH 0 and fluorescent at pH 3.0 (*7*).

Erythrosin: orange at pH 0 and fluorescent at pH 3.6 (*7*).

Esculin: colorless at pH 1.5, blue fluorescent at pH 2 (*1*).

4-Ethoxyacridone: green fluorescent at pH 1.2, and blue at pH 3.2 (*19*).

3, 6-Tetramethyldiaminoxanthone: green fluorescent at pH 1.2, blue fluorescent at pH 3.4 (*19*).

pH 2 to 4

Chromotropic acid: colorless at pH 3.5, blue fluorescent at pH 4.5 (*22*).

Eosin (*C.I. 68*)*:* colorless at pH 2.5, green fluorescent at pH 4.5 (*7;* cf. previous data for eosin).

Fluorescein (*C.I. 766*)*:* colorless at pH 4, green fluorescent at pH 4.5 (*24, 2*).

Magdala Red: purple colored at pH 3.0, fluorescent at pH 4.0 (*31*).

α-Naphthylamine: colorless at pH 3.4, blue fluorescent at pH 4.8 (*19*).

β-Naphthylamine: colorless at pH 2.8, violet fluorescent at pH 4.4 (*19*). This indicator has been described as having a second end point, colorless at pH 3.4 and blue fluorescent at pH 4.8 (*19, 18*).

Phloxine (*C.I. 774*)*:* colorless at pH 3.4 and bright yellow fluorescent at pH 5.0. Some brands vary in behavior.

Salicylic acid: colorless at pH 2.5, blue fluorescent at pH 3.5.

pH 4 to 6

Acridine (*C.I. 788*)*:* green fluorescent at pH 4.9, violet at pH 5.1. The value 5.0 has also been given (*33*).

Dichlorofluorescein: usually colorless at pH 4.0, and bright green fluorescent at pH 6.0 (*18*). The value 6.6 has also been given instead of 6.0 (*8*).

3, 6-Dioxyxanthone: colorless at pH 5.4, blue-violet fluorescent at pH 7.6 (*19*).

Eosin (*C.I. 768*) and *Xylene Cyanol FF* (*C.I. 715*), in ethanol and *n*-butanol: colorless at pH 4.0, green fluorescent at pH 5.0 (*25*).

Erythrosin (*C.I. 772*)*:* colorless at pH 4.0, light yellow fluorescent at pH 4.5 (*18*). The fluorescent color at pH 4.0 has also been stated as green.

β-Methylesculetin: colorless at pH 4.0, blue fluorescent at pH 6.2 (*18, 19*).

Neville-Winther acid: colorless at pH 6.0, blue fluorescent at pH 6.5 (*12*).

Resorufin: yellow fluorescent at pH 4.4 and a weak orange at pH 6.4 (*19*). This indicator sometimes is colorless at pH 4.4 and a stronger orange at pH 6.4, apparently depending upon the product.

Quininic acid: yellow at pH 4.0, blue fluorescent at pH 5.0 (*20*).

Quinine (first end point): blue fluorescent at pH 5.0, violet fluorescent at pH 6.1 (*12, 11, 26, 14*).

pH 6 to 8

Acid R Phosphine: claimed to be useful in the range pH 6.0-7.0.

Brilliant Diazol Yellow: colorless at pH 6.5, violet fluorescent at pH 7.5 (*12*).

Cleves acid: colorless at pH 6.5, green fluorescent at pH 7.5 (*12*).

Coumaric acid: colorless at pH 7.2, green fluorescent at pH 9.0 (*19*).

3, 6-Dioxyphthalic dinitrile: blue fluorescent at pH 5.8, green fluorescent at pH 8.2 (*19*).

Magnesium 8-hydroxyquinolinate: colorless at pH 6.5, golden colored fluorescence at pH 7.5 (*15*).

β-Methylumbelliferone: colorless at pH 7.0, fine blue fluorescence at pH 7.5 (*4, 28, 23*).

1-Naphthol-4-sulfonic acid: colorless at pH 6.0, blue fluorescent at pH 6.5 (*12*).

Orcinaurine: colorless at pH 6.5, green fluorescent at pH 8.0 (*20*).

Patent Phosphine (C.I. 789): for the region pH 6.0–7.0 (*1*).

Thioflavin (C.I. 816): for the region pH 6.5–7.0 (*1*).

Umbelliferone: colorless at pH 6.5, blue fluorescent at pH 7.6 (*30*).

pH 8 to 10

Acridine Orange (C.I. 788): orange colored at pH 8.4, green fluorescent at pH 10.4 (*12, 19*).

Ethoxyphenylnaphthostilbazonium chloride: green fluorescent at pH 9, losing the emission at pH 11.

G Salt: dull blue fluorescence at pH 9.0, bright blue fluorescence at pH 9.5 (*12*).

Naphthazol derivatives: stated to be colorless at pH 8.2, and yellow or green fluorescent at pH 10.0 (*12*).

α-Naphthionic acid: blue fluorescent at pH 9, green fluorescent at pH 11 (*12*).

2-Naphthol-3, 6-disulfonic acid: dark blue fluorescent at pH 9.5, light blue fluorescent at higher values (*13*).

1, 2-Naphtholsulfonic acid: dark blue fluorescent at pH 9.4, changing to a lighter fluorescence at higher values (*14*).

β-Naphthol: colorless at pH 8.6, blue fluorescent at higher values (*14*).

α-Naphtholsulfonic acid: dark blue fluorescent at pH 8.0, bright violet at pH 9.0 (*29*).

β-Naphtholsulfonic acid: dark blue fluorescent at pH 9.0, bright violet at pH 10.0 (*29*).

1, 4-Naphtholsulfonic acid: dark blue fluorescent at pH 8.2, light blue at higher values (*14*).

Orcinsulfonphthalein: yellow colored at pH 8.6, fluorescent at pH 10.0 (*8*).

Quinine (second end point): violet fluorescent at pH 9.5, colorless at pH 10.0 (*12, 11, 26, 14*).

R-Salt: dull blue fluorescent at pH 9.0, bright blue fluorescent at pH 9.5 (*12*).

Sodium 1-naphthol-2-sulfonate: dark blue fluorescent at pH 9.0, bright violet fluorescent at pH 10.0 (*29*).

pH 10 to 12

Coumarin: deep green fluorescent at pH 9.8, light green fluorescence at pH 12.

Eosin BN (C.I. 771): colorless at pH 10.5, taking on a yellow fluorescence at pH 14.0.

Papaverine (oxidized by permanganate): yellow fluorescent at pH 9.5, blue fluorescent at pH 11.0 (*20*).

Schaeffers Salt: violet fluorescent at pH 5.0, green-blue fluorescent at pH 11.0 (*12*).

SS-Acid (sodium salt): violet fluorescent at pH 10.0, yellow at pH 12.0 (*12*).

pH 12 to 14

Cotarnine: yellow fluorescent at pH 12.0, white at pH 13.0 (*12, 11, 26, 14*).

α-Naphthionic acid: blue fluorescent at pH 12, green fluorescent at pH 13.

β-Naphthionic acid: blue fluorescent at pH 12, violet fluorescent at pH 13.

As is evident, certain of the fluorescent indicators listed above exhibit two end points, *e. g.*, quinine. It is emphasized, however, that in some cases, especially the dyestuffs or little-studied indicators of a complex make-up, different brands or slight purity differences may explain the few discordant data of the literature, *e. g.*, α-naphthionic acid, β-naphthylamine, and erythrosin.

In any event, there is much room for additional research in the field of fluorescent indicators. Literally, the fluorescence variation of an organic compound with pH is known in comparatively few instances when it is considered that several thousand fluorescent compounds are known.

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COLLEGE TEACHING LOADS, 1951–52[1]

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Fisher Scientific Company, Pittsburgh, Pennsylvania

For some time interest has been evident among college chemistry-department staff members regarding the clock-hour weekly teaching loads of: (*a*) staff members in schools of various sizes, (*b*) different ranks within a certain range of student body, and (*c*) comparison loads between physical science and nonphysical science staff members, of equal rank, on the same campus.

In at least one instance school administrative officials are of the opinion that the chemistry staff weekly clock-hour teaching load is lighter than that of other departments on the same campus.

In an effort to obtain data designed to yield information on these topics, a questionnaire was prepared and distributed to staff members of approximately 120 schools. At the time of preparation of this report 75 questionnaires have been returned. Cooperation has been very good with only two schools specifically declining to supply the desired data.

No attempt was made to select schools as to size or geographical location. It is quite possible that a more extensive summary covering 200 to 300 schools would have yielded more significant data. It is believed the information submitted on the returned questionnaires is of considerable significance and represents a trend regarding clock-hour loads and teaching credits.

SUMMARY OF INFORMATION

The undergraduate enrollment of the schools included in this study ranged from 400 to 19,000 students. The undergraduate chemistry enrollment (courses × students) ranged from 57 to 3384, and the graduate chemistry enrollment varied from 0 to 280. With such an obvious wide range, in values, it was quite impossible to prepare a simple summary. Thus the data were divided into four different classes (groups), based upon total undergraduate enrollment of schools concerned. Table 1 shows the arbitrary subdivision used in this classification.

TABLE 1

Group	*Range of undergraduate enrollment*	*No. of schools included*
A	400– 1,000	18
B	1,001– 3,000	22
C	3,001– 9,999	25
D	10,000–19,000	10

Table 2 gives information relative to the number of schools in each group which have graduate students in chemistry (M.S. or Ph.D. candidates) together with the total number of such graduates registered during the school year 1951–52. Information regarding the relative load of chemistry department staff with respect to man-hour teaching load of other nonphysical science departments on the same campus is also included. (In some cases the information was submitted in "quantitative" nature, while in the majority of reports it was merely "qualitative.")

TABLE 2

Group	*No. schools with graduate students*	*Total graduate students*	*No. schools whose teaching loads of chem. staff, with respect to those of nonphysical science staff, are:*		
			Heavier	*Equal*	*Lighter*
A	3	37	12	3	3
B	14	517	11	10	1
C	22	1,839	6	12	5[a]
D	10	1,374	0	8	2

[a] Two schools declined to release information concerning teaching loads in different departments.

In certain schools (primarily in the A and B) groups administrative officials are concerned with the "student-teacher load," more than with the "clock-hour load" of the teaching faculty. The "student-teacher load" is computed by multiplying the class registration by the contact hours of the professor. One example is cited in which the chemistry staff averaged 15.9 clock-hours contact per week, while a nonscience department averaged 15.0 contact hours per teacher. When "student-teacher loads" of these departments were computed, it was found the chemistry staff had a value of 216 while the nonscience staff had a value of 360. This low figure for the chemistry staff is due to limited enrollment in advanced courses. Irrespective of student load, it is obvious that time of preparation and presentation of material for a class of 10 to 12 students is equal to that involved for a class of 25 or 30. On the other hand, one must remember that those schools which derive a major portion of their income from student tuition and fees must obviously be "class-size" conscious.

Such experiences as are illustrated herein are not as evident in the larger state-supported or heavily endowed institutions.

Table 3 presents data concerning the "laboratory clock-hour: credit-hour" distribution in the schools included in this study. In certain schools the full-time staff does not teach laboratory and thus information on this item was not available.

Table 4 gives a detailed breakdown, by groups, of the

[1] Presented at the 122nd Meeting of the American Chemical Society, Atlantic City, New Jersey, September, 1952.

TABLE 3

Group	Laboratory clock-hour : credit-hour ratio				
	1:1	2:1	4:3	3:1	3:2
A	5	7	1	3	1
B	7	6	3	3	2
C	10	5	2	3	1
D	6	4	0	0	0

average weekly total clock-hours as well as the average weekly clock-hours spent in laboratory by each of the four full-time academic ranks considered.

Some of the schools included in the study do not have chemistry staff members holding all four academic ranks. (For example, some have no instructors while others do not have associate professors of chemistry.) In other schools certain members of the full-time staff do not teach laboratory. Thus there is considerable variation in the number of institutions concerned in the determining of the average values appearing in Table 4. In order to indicate the number of schools involved in computing the averages shown in Table 4, the number in parenthesis "( )" above each average value is the number of institutions involved in obtaining the average.

Certain schools consider the intentional reduction in clock-hour teaching load as an adjustment for carrying out as well as directing graduate research. Thus they do not give additional time allowances for either supervising laboratories or directing research. On the other hand, certain schools give special reduction in teaching load (clock-hours) for: (*a*) carrying a portion of the administrative duties of the department, (*b*) laboratory supervision, and/or (*c*) directing graduate research. In still other schools an "elastic" policy within the chemistry department permits the adjustment of specific individual's teaching loads to accommodate these and other duties. Table 5 gives a cross-section of data related to these items. The number of schools in each group which provided for such factors is indicated in this table.

In several of our larger schools, all laboratory instruction is conducted by graduate-assistant instructors. Table 6 shows the weekly clock-hour teaching load of staff members at schools where the full-time staff does not teach in laboratory. Data do not show teaching load in the event a specific rank full-time teacher *does* teach laboratory in addition to his lecture-recitation work.

TABLE 5

Group	Reduction of certain academic teaching loads for other duties			
	Administrative duties	Research direction[a]	Supervision of laboratories	Elastic dept. policy
A	8	1	1	3
B	13	5	1	1
C	17	8	4	2
D	7	1	5	5

[a] The reduction in teaching load for direction of graduate research is quite variable. One school permits one-half hour reduction per week per student up to a maximum of 6 students (3 hours total maximum reduction), while another school allows one hour reduction per student up to a maximum of 5 students. This policy represents a definite trend.

TABLE 6

Group	Instructor	Asst. prof.	Assoc. prof.	Full prof.
		(1)[a]	(1)	(1)
A	0	7	7	6
	(3)	(5)	(6)	(6)
B	12	11.2	10.5	8.8
	(3)	(3)	(3)	(6)
C	11.8	7.7	7.0	7.3
	(5)	(5)	(5)	(5)
D	8.0	7.2	7.2	6.8

[a] Numbers in parentheses above each teaching load value indicate number of institutions considered in determining the average value.

DISCUSSION

Table 1: Data appearing in Table 1 indicate that a fair distribution of size of groups has been obtained by separation at the enrollment basis indicated. Group A includes four girls' schools. Group B includes three schools for colored students. Group C contains five state universities, while Group D included six state universities and one large Canadian school.

Table 2: This table indicates several factors which would be expected. (1) Chemistry staff teaching loads at smaller schools are generally heavier than those at the larger institutions where graduate assistants are available for assisting in laboratory instruction. The need for devoting more time (clock-hours) in laboratory in order to receive credit hours of instruction is given as a partial explanation of the "overload" indicated. Some school administrative officers are of the opinion that laboratory instruction is not as "strenuous" as classroom and does not merit hour-per-hour credit.

(2) The institutions with larger enrollments have a teaching (clock-hour) load comparable with that of staff members in nonscience departments.

TABLE 4

Group	Weekly load: rank (full-time staff (average))							
	Instructor		Asst. prof.		Assoc. prof.		Full prof.	
	Total	Lab	Total	Lab	Total	Lab	Total	Lab
A	(5)	(5)	(14)	(11)	(10)	(7)	(16)	(10)
	17.5	10	15.5	10.0	14.1	9.2	15.4	10.3
B	(15)	(9)	(19)	(13)	(20)	(14)	(22)	(14)
	16.1	10.5	15.1	9.3	14.9	9.2	14.1	9.0
C	(22)	(14)	(24)	(16)	(24)	(18)	(23)	(15)
	14.5	8.3	12.9	7.2	12.7	7.0	11.7	6.1
D	(9)	(4)	(10)	(4)	(10)	(4)	(10)	(4)
	10.3	8.7	9.4	6.0	9.3	6.2	9.1	6.2

Table 3: This contains information of considerable interest.

(1) A wide range of clock-hour: credit-hour credit exists in our college laboratory teaching by full-time chemistry staff members. This is quite independent of the size of the institution concerned.

(2) Approximately one-quarter of the schools in the smallest enrollment group grant their staff members hour-to-hour credit in laboratory, while several schools of approximately ten times the student body require staff members to spend two to three clock-hours in laboratory for one hour teaching credit.

(3) It is rather surprising to learn that some institutions having enrollments above 3000 students require three clock-hours in the laboratory for one of credit. In fact, some schools with enrollments above 5000 are requiring two hours of laboratory work for one of credit.

(4) The clock-hour: credit-hour ratios of 3:2 and 4:3 are not popular in college chemistry laboratory instruction.

Table 4: Attention is called to the following information obtainable from data appearing in Table 4:

(1) The clock-hour teaching load of full-time staff members in smaller schools is greater than that for equal rank at larger schools.

(2) The magnitude of the decrease in teaching load with increasing academic rank is greater in smaller schools than in larger academic institutions.

(3) Certain of our larger schools maintain laboratory instruction by all academic ranks of full-time faculty members. It is gratifying to note that highly experienced faculty at certain schools are active in laboratory instruction.

Table 5: It is believed the information presented in Table 5 is self-informative. The "elastic department policy" is one in which the departmental head may reduce the teaching load of certain staff members to provide for such items as: (*a*) government grants, (*b*) research fellowships, and/or (*c*) administrative or other duties.

Table 6: Information included in this Table shows:

(1) Practically all of our smaller schools require laboratory instruction by all ranks of full-time teaching personnel.

(2) The teaching load of nonlaboratory-teaching lower academic rank personnel is heavier than that of higher ranking staff members in the intermediate size schools (Groups B and C), while it is fairly constant in the schools with largest enrollment (Group D).

CONCLUSION

Information contained in this study covering teaching loads of chemistry department full-time staff members may serve as a satisfactory check and comparison basis for individual schools whose data is included as well as those whose data is not included in this survey.

RELIGIOUS PERSPECTIVES OF COLLEGE TEACHING

In the pursuit of wisdom the teacher of science must find the opportunity to convince the student that beyond the areas covered by science and scientific conclusions, beyond the testimony of history, there are areas of truth which supplement those of knowledge to yield *sapientia*. These embrace art, literature, philosophy, and religion. They include emotional and symbolic conclusions which require discipline and training no less than those activities practiced in the laboratories, require a critical appreciation that can estimate the ultimate work of an art, of music, of literature, of all forms of effort that can lead to valid emotional reactions. The emotions will need to be fortified by a disciplined capacity for ethical, philosophical, and religious conclusions. The urgent need is not for the science specialist but for the liberally educated man. Such a man will recognize the necessity for a return to unity in place of a supposed dichotomy between scientific techniques on the one hand and moral idealism and religious faith on the other. There is no necessary reason why a scientific world civilization need be sundered from a universal religious faith. The rationalizm that is necessary to the ordering of the material world in the minds of men need not be divorced from a religious approach ordering human life toward spiritual ends. Indeed, it may be urged that a fusion of the two into a common unity is the signal need in the world of today to resolve the stresses and strains. Unless we can ennoble the material realities that are available to us with the spiritual realities that are even more fundamental the outlook is dark indeed. It is the age-long struggle for primacy between the material and the spiritual. Now, when man's capacity for control over the material through science is becoming ever more potent, it is even more essential that he pursue with equal intensity the principles of a spiritual order. Somehow or other the teacher of science must communicate in his teaching, in his work, in his life, the truth that our physical universe can go down into physical death unless we can at the same time make of it a sacramental universe. In the dark days of our time there will be many opportunities for all of us to trace out all the implications of the word "sacrifice." Death and wounds, hurt and pain, economic loss, long hours of toil. Something will have escaped us if we fail to recognize, beyond all the material considerations, something in the word that goes back to its Latin roots: *sacrum facere;* in making sacred our daily effort and toil in the laboratory, the lecture hall, the factory, or workshop we can achieve the unity of life and the faith in the future that are essential for well-being. "Does the road wind uphill all the way? Yes, to the very end."

Hugh S. Taylor

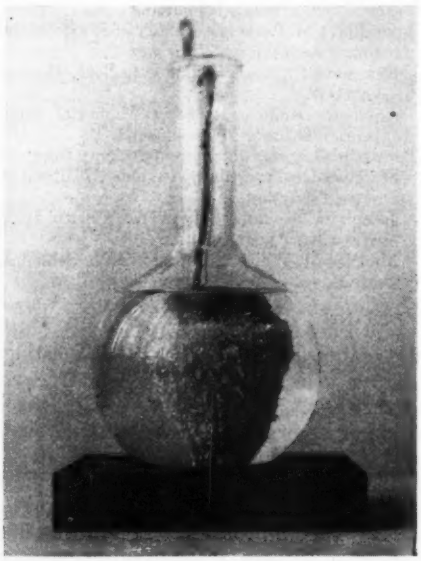
SILVER TREES

DAVID I. GLEIM
Dickinson College, Carlisle, Pennsylvania

TO DEMONSTRATE a displacement reaction, I know of no simpler or more spectacular method than the formation of a silver tree.

If a loose coil of fine copper wire is inserted into a 500-ml. Florence flask containing 10 g. of silver sulfate dissolved, by heating, in 480 ml. of distilled water and 20 ml. of concentrated sulfuric acid, a silver tree will start to form immediately and in from ten to fifteen minutes will occupy a large volume of the flask. This growth is quite fragile and much handling will cause it to break from its wire support.

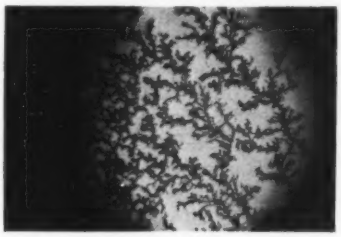
If the finer details of the structure are to be observed one must resort to the microscope. On a clean microscope slide place a drop of concentrated sulfuric acid and dissolve in it, with the aid of a little heat, a very small quantity of silver sulfate. Too high a concentration of the silver salt will produce secondary effects. A small piece of a metal below hydrogen in the electromotive series, as antimony, bismuth, or copper, is allowed to fall on the drop, which is then covered with a cover glass. With the exception of bismuth, a growth will form almost immediately. When bismuth is used as the displacing agent, the sulfuric acid must be diluted with a small drop of water. Silver nitrate dissolved in water



Typical Silver Tree in Approximately Five Minutes

will give results similar to those with silver sulfate in sulfuric acid.

Accum, in his "System of Theoretical and Practical Chemistry," published in 1814, describes on pages 31 and 32 of volume 2 several methods for the production of silver trees or "Arbor Dianae." In one procedure, which he claimed succeeds better than any of the others, the electrolyte was a mixture of silver and mercuric nitrates, while the displacing agent, mercury, was introduced in the form of an amalgam with silver. It is interesting to note that almost 150 years ago



Ag_2SO_4 in H_2SO_4 + Bi

chemists were aware of equilibrium in reactions.

Accum's procedure was carried out by the writer. The growths produced remind one of an aerial view

Dilute $AgNO_3$ + Bi

of a wooded area, with here and there a taller tree standing out amongst the others. Their height varies from one eighth to one quarter of an inch and they are

Dilute $AgNO_3$ + Cu

remarkably sturdy compared to trees formed by ordinary displacements. As a demonstration the procedure has no value.

Report of the NEW ENGLAND ASSOCIATION OF CHEMISTRY TEACHERS

OFFICIAL BUSINESS

267th MEETING

The 267th meeting of the NEACT was held on October 18, 1952, at St. Paul's School, Concord, New Hampshire. The Eastern Association of Physics Teachers met simultaneously in a joint meeting. The morning program consisted of two addresses. The first was presented by Kenneth A. Blaine, a leather tanner andchemist at the International Shoe Company, Manchester, New Hampshire, who outlined the history, chemistry, and present status of leather tanning in an interesting talk, enlivened with amusing anecdotes.

Richard D. Wells, research director of Bates Manufacturing Company, Lewiston, Maine, was the second speaker, whose topic was "Science education for today." His original thinking and able expression won acclaim from the group. His address appeared in the February issue of THIS JOURNAL.

After an excellent luncheon in the Alumni Hall, the afternoon session concluded with a lecture-demonstration by Karl Woodcock, head of the Physics Department of Bates College, Lewiston, Maine. Dr. Woodcock's topic was "Microwaves." He presented analogies of microwaves with light, using special equipment developed for easy understanding of the underlying principles.

Business Meeting. President Dorothy W. Gifford conducted the business meeting. The Secretary reported that the following new members had been elected since the summer conference:

Harold Finegold, Instructor in Chemistry, Boston University, Boston, Massachusetts.
Donald F. Hornig, Professor of Chemistry, Brown University, Providence, Rhode Island.
Margaret M. Parks, Associate Professor of Chemistry, University of Rhode Island, Kingston, Rhode Island.
Eugene G. Rochow, Associate Professor of Chemistry, Harvard University, Cambridge, Massachusetts.
J. H. Shrader, Eastern Nazarene College, Wollaston, Massachusetts.
Newell N. Toothaker, Teacher of Chemistry, Sanford, Maine.

President Gifford read a summary of the financial statement of the 14th Summer Conference, an abridged version of which follows:

Receipts:		
Registrations	$ 782.00	
Hostelry	1744.50	
Exhibitors	125.00	
Total Receipts		$2651.50
Disbursements:		
Hostelry	$1761.22	
Printing, postage, etc.	369.41	
Dues to NEACT	92.00	
Miscellaneous	93.97	
Refunds	112.00	
Total Expenses		$2428.60
Cash on Hand		$ 222.90

President Gifford announced the following appointments and committees:

Assistant Editor of the News Letter: Eva M. Ruggli, 67 Dana Street, Cambridge 38, Massachusetts.
Assistant Editor of the Report: William G. Chace, Lowell Textile Institute, Lowell, Massachusetts.
Representatives of the NEACT on the New England School Science Council: Dorothy W. Gifford, Lincoln School, Providence, Rhode Island, *ex officio;* Helen W. Crawley, Walnut Hill School, Natick, Massachusetts; Everett F. Learnard, Norwood High School, Norwood, Massachusetts.
Subcommittees of the NEACT Chemical Education Committee:
(1) To draw up a syllabus for nursing school students:
Margaret M. Parks, University of Rhode Island, Kingston, Rhode Island, *Chairman*
Raymond E. Neal, Simmons College, Boston, Massachusetts.
Anne R. Kelley, Roxbury Memorial High School (Girls), Boston, Massachusetts.
(2) To establish criteria for judging science fairs:
Standish Deake, Milton Academy, Milton, Massachusetts, *Chairman.*
Joseph H. Rohloff, Nelson W. Aldrich High School, Warwick, Rhode Island.
Maurice M. Whitten, Lewiston High School, Lewiston, Maine.

268th MEETING

On December 13, 1952, the 268th meeting of the NEACT was held at Boston College, Chestnut Hill, Massachusetts, as a joint meeting with the Eastern Association of Physics Teachers and the New England Biological Association. About 200 persons attended. Father John Tobin, S. J., chairman of the Physics Department of Boston College, presided at the morning session. The first speaker was Dr. T. E. McCarthy of the Boston College Chemistry Department who spoke on "Biology, chemistry, and physics in the medical laboratory." He called attention to some misconceptions concerning the status of employees in medical laboratories, assuring the audience that technologists are not underpaid, nor always assigned routine work. He outlined the various classifications of workers in

the field, as recognized by the American Society for Clinical Pathologists, and the training required for each type of appointment. There are positions available that require only a high school education and others that require the Ph.D. degree, with all gradations in between. The work is varied also. Besides routine tests, that make up the bulk of the work for the lower classification of worker, there are among the more advanced assignments, vitamin assays, pregnancy tests, glucose tolerance tests, determination of calcium by use of the flame spectrophotometer, all as an aid to the clinician in his diagnosis. A great deal of research is also undertaken by those qualified to do it. Dr. McCarthy's remarks were especially helpful to teachers who need accurate knowledge of job opportunities because of their vocational guidance work.

The remainder of the morning session was devoted to a panel discussion on "Judging high-school science fairs." Raymond F. Scott, Rindge Technical High School, Cambridge, Massachusetts, opened the discussion by defining a science fair as a collection of student-prepared science exhibits and by stating that the purpose of a fair was to discover potential scientists and to develop scientific habits and interests. He discussed in detail the criteria for judging that have been evolved by the committee in charge of judging of the Massachusetts State Science Fair, under the leadership of Dr. Albert Navez of the Newton, Massachusetts, school system.

Malcolm Campbell, president of the New England Biological Association and a teacher at the Dorchester High School for Boys, Boston, Massachusetts, was the second speaker on the panel. He presented his philosophy in directing students in their preparation of exhibits and emphasized fair play on all sides. Elbert C. Weaver, Phillips Academy, Andover, Massachusetts, concluded the discussion with suggestions of exhibits which he, as a judge, would like to see among student presentations. He prefers projects which require measurement, as distinct from poster exhibits, that may have great dramatic appeal but little science. Among his suggested projects were the following: development of pH indicators from coloring matter on postage stamps or from natural vegetable sources; determination of the characteristics of the local water supply and the methods used to make is potable; studies of electroplating processes; determination of the efficiency of various commercial thermal insulators, such as frozen food containers; and the analysis of the construction of various household appliances. He hopes for the day when the student-exhibitor will present as part of his exhibit, his day-to-day notebook of measurements and observations as well as a working model of his device. Only if the standards of judging are changed will students feel it advantageous to offer such "homely" exhibits, but the scientific achievement of the fairs will be enhanced.

Fletcher Watson of the Graduate School of Education, Harvard University, made an appeal to the teachers present to draw up a list of personal and professional characteristics of the "outstanding teacher." The composite being resulting from an appraisal of suggestions will serve as a model to guide the committee of the American Academy of Arts and Sciences in their selection of candidates for the annual award now being made to outstanding science teachers in New England.

The three associations met separately in the afternoon session. Father James Devlin, S. J., of the Physics Department of Boston College, spoke before the NEACT on flame spectroscopy. He took his audience through the maze of molecular and atomic structures that are revealed by emission and absorption of energy in the form of band or line spectra. He demonstrated the optical system employed in the analysis of spectra and gave an inkling of the difficulty of interpreting band and line spectra. He concluded his remarks by referring to some applications of spectroscopy as an analytical tool in the metal industries.

Business Meeting. The President, Dorothy W. Gifford, Lincoln School, Providence, Rhode Island, conducted the meeting. The secretary welcomed the following members to the Association. This list brings the number of new members this year up to 47.

Henry A. Bent, Instructor in Chemistry, University of Connecticut, Storrs, Connecticut.
Brother Andrew Arthur, F. M. S , Teacher of Chemistry, Central Catholic High School, Lawrence, Massachusetts.
Oliver L. I. Brown, Chairman of Chemistry Department, Connecticut College, New London, Connecticut.
William L. Davidson, Instructor in Chemistry, Thomas Jefferson High School, Elizabeth, New Jersey.
Maxwell M. Erskine, Instructor in Chemistry, Edward Little High School, Auburn, Maine.
Morton A. Fineman, Assistant Professor of Chemistry, Providence College, Providence, Rhode Island.
Theodore T. Galkowski, Assistant Professor of Chemistry, Providence College, Providence, Rhode Island.
Norman G. Mills, Teacher of Chemistry, South Hadley High School, South Hadley, Massachusetts.
John Osterman, Head of Chemistry Department, Hingham High School, Hingham, Massachusetts.
Sister Jeanne de Florence, A. S. U., Science Teacher, St. Joseph Academy, Salem, Massachusetts.
Sister Mary Adele, Teacher of Chemistry, St. Ann's High School, Gloucester, Massachusetts.
Sister Mary Nativitas, S. S. J., Science Teacher, St. Mary's High School, Brookline, Massachusetts.
Sister Mary Philemon, R. S. M., Instructor, Salve Regina College, Newport, Rhode Island.
P. Conant Voter, Chairman of the Chemistry Department, Middlebury College, Middlebury, Vermont.
Lewis T. Voultos, Instructor in Chemistry, Middleboro Memorial High School, Middleboro, Massachusetts.
Eleanor R. Webster, Instructor in Chemistry, Wellesley College, Wellesley, Massachusetts.
Phyllida M. Willis, Assistant Professor of Chemistry, Wellesley College, Wellesley, Massachusetts.

15th Summer Conference. President Dorothy W. Gifford announced that the Executive Committee has accepted the invitation from Bowdoin College to hold the 15th Summer Conference on its Brunswick, Maine, campus during the week beginning August 23, 1952. John E. Cavelti, Head of the Chemistry Department of Allegheny College, Meadville, Pennsylvania, and Guy F. Burrill, Keene High School, Keene, New Hampshire,

will serve as co-chairmen. Anyone wishing further information about the summer conference is invited to write to the Secretary of the conference, William O. Brooks, Technical High School, Springfield, Massachusetts.

The secretary presented a résumé of the activities of the Subcommittee on Chemistry of the School and College Study of Admission with Advanced Standing. This study has been sponsored by eleven liberal-arts colleges and the Massachusetts Institute of Technology, to seek a method of granting advanced standing to exceptionally well prepared students from secondary schools at the time of their admission to college. It was voted that the New England Association of Chemistry Teachers express its enthusiastic support of the work of this subcommittee.

To the Editor:

The article entitled "The reading difficulty of textbooks for high-school chemistry" [J. CHEM. EDUC., **29,** 629–31 (1952)] well illustrates the dangers of oversimplifying a complex situation. The temptation to reduce a concept of many parameters to a single numerical coefficient is strong in human nature, but one must keep in mind that the end result is no more sound than are the simplifying assumptions made along the way. In this article the implicit assumption is that the "reading difficulty score," compiled from objective criteria (words per sentence, syllables per word, and frequency of personal references), is a real measure of what we identify subjectively as reading difficulty. No validation of this assumption occurs in the article; in fact, one may suspect that the nature of the concepts presented and their manner of presentation are often the major components of reading difficulty in scientific material—these are ignored by the authors.

I submit that, in order to avert misunderstandings, coefficients purporting to represent many-factored concepts be designated by neutral terms until their significance is established. Let us call the "reading difficulty score," for example, "the Zeta Index of Reading Difficulty" until we are reasonably certain that it encompasses the major parameters of reading difficulty. An obvious method for validating the authors' coefficient is to establish a correlation of the coefficient with the collective judgment of experienced teachers of high-school chemistry with respect to the difficulty of each of the twenty-two textbooks used in the study. Internal validation such as that referred to in Conclusion 6 of the article is circular and therefore meaningless.

Until the real significance of the coefficient is established, Conclusion 7, that "reading difficulty of the popular textbooks in the field of chemistry is not likely to discourage students from enrolling in chemistry," lacks foundation and may have a deleterious effect on thinking about the very real problem of decreasing chemistry enrollments. In the words of J. W. N. Sullivan, "It is much easier to make measurements than to know exactly what you are measuring."

WILLIAM HERED
CALUMET CENTER, INDIANA UNIVERSITY
EAST CHICAGO, INDIANA

To the Editor:

Chemistry teachers and authors of chemistry textbooks can take heart if the study of Mallinson, Sturm, and Mallinson [J. CHEM. EDUC., **29**, 629–31 (1952)] is reliable. Their study shows that all except one of twenty-two high-school textbooks in chemistry have a reading-difficulty level at least one or two grades below the reading level of the intended audience. They infer that the lower the reading-difficulty level, the better.

Their conclusions are based on a modification of the Flesch formula in which from five to eight 100-word samples were studied, depending on the length of the book.

Two disturbing factors are obvious: (1) Professor Flesch ["How to Test Readability," Harper and Brothers, New York, **1951**, p. 1] recommends 25 to 30 100-word samples in order to rate a book. The modification of the Flesch formula possibly has a reliability factor of about 0.2. (2) The subject matter of elementary chemistry varies widely in degree of difficulty. Possibly the wide range between the reading-difficulty scores of the samples of a given textbook is connected with subject-matter.

In view of these two points, it would be interesting to hear from the authors as to whether or not they found these scores reproduceable for a given chemistry textbook.

The implications of the study are also important, as Professor Hered points out in another manner. Are we challenging our ablest readers or are we losing their interest in chemistry by oversimplification? Since many of our chemistry students are in the college-preparatory group in high schools, should they not get a taste of mature writing? On the other hand, very little has been done that cannot be done better. This generalization certainly applies to the writing of chemistry textbooks.

Professor Hered introduces a subjective factor into the picture (judgment of experienced high-school chemistry teachers) which may be based upon intangibles not connected with chemistry textbooks at all. For example, the personality of a publisher's representative may influence a teacher's views about a book. Also, the book that is best for one teacher may not be best for another teacher, regardless of reading-difficulty level. So Professor Hered's words of caution are obviously timely.

ELBERT COOK WEAVER

PHILLIPS ACADEMY
ANDOVER, MASSACHUSETTS

To the Editor:

As you know, electrons in the highest energy levels of the atoms, those that cause chemical combinations, are called *valence electrons.*

I should like to suggest the term *valentrons* for the expression *valence electrons.*

The above-proposed name has the advantage of being simple, easy to understand, and, I think, also applicable to international nomenclature.

J. V. AZCUENAGA-CHACON
PONTIFICIA UNIVERSIDAD CATOLICA JAVERIANA
BOGOTA, COLOMBIA

To the Editor:

It follows from the conception of atomic weight that weights of different elements numerically equal to their respective atomic weights contain the same number of atoms. (With weights expressed in grams, this means that the gram atoms of different elements contain the same number of atoms.)

But this fact may not at first be realized by the beginner in chemistry though none of the textbooks known to the author, apparently taking it for granted, has given expression to it. It is, however, thought that the enunciation of this fact supported by a mathematical derivation in textbooks and/or classroom lectures will greatly help in getting the fundamentals of the atomic theory clearly understood by the beginner in the subject. Avogadro's hypothesis might then be compared and contrasted with it at the appropriate stage to great advantage. The mathematical derivation could be given in some such manner as follows:

Consider any two elements A and B. Let the atomic weight of A be x, and the atomic weight of B be y. Then

$$\frac{x}{y} = \frac{\text{Wt. of 1 atom of A}}{\text{Wt. of 1 atom of B}}$$

since, by definition of atomic weight,

$$x = \frac{\text{Wt. of 1 atom of A}}{\text{Wt. of 1 atom of hydrogen}}$$

and

$$y = \frac{\text{Wt. of 1 atom of B}}{\text{Wt. of 1 atom of hydrogen}}$$

Let the absolute weight of 1 atom of A be m gram, and the absolute weight of 1 atom of B be n gram. Then

$$\frac{x}{y} = \frac{m}{n}$$

Or

$$\frac{x}{m} = \frac{y}{n}$$

But

$$\frac{x}{m} = \text{Number of atoms in } x \text{ grams, or gram atom of A}$$

and

$$\frac{y}{n} = \text{Number of atoms in } y \text{ grams, or gram atom of B}$$

Therefore weights of A and B numerically equal to their atomic weights, *i. e.*, x and y grams respectively, contain the same number of atoms.

The foregoing is only submitted in the belief that it will be of some educational interest.

C. N. NAIR

ASSAM OIL COMPANY, LTD.
DIGBOI, ASSAM, INDIA

To the Editor:

There is I think a better and simpler method of dealing with the student questions on the Hittorf method of determining transport numbers than that given by Arthur L. Levy [J. CHEM. EDUC., **29,** 384 (August 1952)].

Faraday's laws express the fact that in the transfer of electricity through a solution the quantity of matter associated with a given quantity of electricity is dependent only on the nature of the matter, including its valency state; concentration is an irrelevant factor. In the Hittorf method of determining transport numbers we attempt to find the proportion of electricity carried by the positive and negative ions by measuring the change in matter around the electrodes. It should therefore be clear that it is the *total mass change* which must be measured and not a concentration change. This may be emphasized by drawing the familar Grotthus type diagram and putting in the dividing lines for the anode and cathode compartments in various places. It will be seen that concentration changes will enable transport numbers to be calculated only if equal volumes are removed from each compartment. Such a restriction makes nonsense of any expression involving concentrations.

In my experience *concentration* is a difficult concept to students who often fall into error by not using the concept where it should be introduced. If this experience is general, then it behooves teachers to be careful to make clear when the concept is relevant and when irrelevant.

L. H. ANGUS

BERKHAMSTED SCHOOL
BERKHAMSTED, ENGLAND

● A TEXTBOOK OF QUANTITATIVE ANALYSIS

Andrew Patterson, Jr. **and** ***Henry C. Thomas,*** **Associate Professors of Chemistry, Yale University. Henry Holt and Co., New York, 1952. xii + 500 pp. Illustrated. 14.5 × 21.5 cm. $4.25.**

THIS book is intended as a textbook for a first course in quantitative analysis. It has been written as a "teaching text" and "not a compendium of laboratory procedures." The authors have attempted to develop the subject matter in a reasonable and logical manner, and in many instances in great detail. They feel that the student can acquire an appreciation of this logic and reasoning only by starting at the very beginning of the book and reading it consecutively page by page. This manner of presentation of material, I would say, is the novel feature of the book.

Because the material is presented in more or less the order that the student needs the information, some unusual arrangements of material result. As examples illustrating this point the following instances are listed: First, in Chapter 2, entitled Volumetric Methods, Titrations with Permanganate, the following topics occur together in this chapter on volumetric analysis: (1) Titrations and standard solutions, (2) Analytical balance, (3) Technique of weighing, (4) Apparatus, (5) Accuracy and precision, (6) Titrations with permanganate. Second, the discussion of the balance and weighing which is begun in Chapter 2 is supplemented and completed in Chapter 6, Procedures Used in Gravimetric Analysis, 20 pages being devoted to weight calibration and a discussion of weighing errors. Third, although the permanganate titration is discussed in Chapter 2, the theoretical treatment of oxidation-reduction together with the various experimental oxidation-reduction titrations other than permanganate are found in Chapter 5, after treatment of acidimetry in Chapter 3 and of precipitation and complex-forming titrations in Chapter 4. However a very good index and the use of topic headings at the top of the pages make it possible to find the material sought without difficulty.

The 100 pages devoted to gravimetric analysis follow the 200 pages on volumetric analysis. Included in the section on gravimetric analysis are discussions on the preparation and properties of precipitates, 10 pages on precipitation of inner-complex compounds, a chapter on procedures and techniques used in gravimetric analysis, and a chapter on various chemical separations such as are used in brass, silicates, etc.

Colorimetry, photoelectric colorimetry, electrochemical analysis, and polarography are discussed in a chapter, Some Physical Methods of Chemical Analysis. Forty-five pages have been allotted to the first three topics and three pages to the last.

The theoretical discussions have been written in considerable detail and are ample in scope for a textbook on elementary quantitative analysis. Concise laboratory procedures, accompanied by notes, follow the theoretical discussions in the various chapters. The procedures and notes are printed in bold-face type to facilitate reading. Unfortunately, the formulas in these sections look somewhat odd because the subscript numbers are not in bold-face type although the symbols are. An ample supply of standard procedures makes a choice available.

About 90 problems are listed throughout the book with about half of them at the end of the chapters on permanganate and acidimetric titrations. A few of the first problems have answers but most are without. A separate four-page pamphlet listing formula weights, gravimetric factors, atomic weights, and a four-place logarithm table is available to accompany the text. Calculations in the permanganate chapter are explained on the titer basis. The concept of calculations by the normality system is introduced in the chapter on acidimetric titrations which follows immediately. Apparently this has been done for expediency as well as to instruct the student in the use of both methods of calculations.

The book has been carefully planned and clearly written. However, it should be mentioned that both molecular and ionic equations have been used rather interchangeably throughout the text. Also the appearance of some of the figures would be much improved if the originals had been drawn with the thought in mind that there was to be reduction in size when printed. On the whole the book is a good one and will warrant careful examination by those who are in the market for a good teaching text.

REX J. ROBINSON

UNIVERSITY OF WASHINGTON
SEATTLE, WASHINGTON

● INTRODUCTORY GENERAL CHEMISTRY

John E. Cavelti, **Professor of Chemistry, Allegheny College, Meadville, Pennsylvania. The Blakiston Co., New York, 1952. xi + 423 pp. 50 figs. 9 tables. 16 × 23.5 cm. $4.50.**

DR. CAVELTI proposes "to induce students to consider the subject as one which is assimilated as readily as the various branches of the humanities, as interesting as they are, and as significant for an understanding of the temper of modern times... The aim has been to write a readable book . . .[for] students not necessarily intending to make chemistry their life work."

The first chapter, Background of Chemistry, is followed by what might be called a 'tool" chapter given to measurements and their units. The concluding third of this chapter gives

"once for all" attention to the scientific method. The sequence, thereafter, includes: Oxygen, Hydrogen, Gases, Theory, (Atoms and Molecules), Oxygen-hydrogen Compounds, The Atmosphere (Nitrogen), Atomic Structure (two chapters), Compound Formation, Energy, Solids, Solutions, Ions, Electron-Proton Reactions, Rates and Equilibria, Extremes of the Periodic System, Salt and Sea Water, Sulfur Phosphorus, Metals (Iron and Aluminum), Magnesium and Calcium, Carbon and Silicon, Organic Chemistry, and last, The Rest of the Periodic System.

The author evidently has little faith in Conant's and other leading scientists' emphasis of the scientific method as the unique contribution of chemistry to the program of general education. The text is "readable," as promised, but too frequently follows the pattern of "I am telling you" in a more or less deductive manner rather than leading the reader to share in the adventure of sleuthing the evolution of the idea.

This first edition has failed to provide a number of desirable teaching aids. There is a scarcity of subtopics within the chapters. This is especially noticeable in Chapter one. The questions are, all too often, of the memory type; references at the chapter ends are not adequately specific; diagrams and illustrations are not numbered, and vocabulary help is deficient. One appendix provides numerical problems keyed to the individual chapters and the index is generous. Perhaps a later edition may give attention to the oversights mentioned.

For teachers who consider chemistry's job done when it has added its parcel of information to the problem of general education this book has much to offer. For clarity of exposition, for felicity of illustration and coherence, and originality of organization it merits a first-line rating.

B. CLIFFORD HENDRICKS

Longview Washington

• SEMI-MICRO METHODS IN ORGANIC CHEMISTRY

Ernest R. Kline, **Associate Professor of Chemistry, University of Connecticut. Burgess Publishing Co., Minneapolis, 1947. v + 225 pp. 38 figs. 13.5 × 21.5 cm. Offset. Spiral bound. $2.25.**

This book has been used by the author in his own classes for a number of years and is now for the first time being offered to other schools for adoption. It is designed to help the student conduct his experiments on a small scale of operations, thus effecting considerable savings in material and reducing the danger hazard to a minimum. The small scale of operations makes it possible to accommodate larger sections in the laboratory without undue crowding.

The collection of laboratory procedures, according to the author, attempts to achieve two additional ends. The first is to use the laboratory exercises as a means of impressing upon the student the significance of the equations which he finds so profusely spread upon the pages of his textbook, and, second, to provide training in semimicro methods during the first year of organic chemistry as a subsequent aid in the course in organic qualitative analysis.

The experiments included would require the usual semimicro equipment and some special pieces that could be provided by a glass blower of average ability. The book contains more than the average amount of theoretical and explanatory material. All preparations included are used to illustrate basic principles or operations which are discussed in turn in some detail. Items so discussed and included are typical replacements, oxidation, reduction, dehydrogenation, molecular rearrangement, etc. This should be a valuable feature for those instructors who prefer to use student laboratory experiences as a supplement to teaching chemical information, as well as mastering useful manual techniques.

Several experiments are included that illustrate well-known named reactions. More than usual emphasis is placed on basic organic laboratory operations and qualitative techniques. The pages are reproduced from author-prepared originals and, while easily discernible, could be improved in places by better reproduction methods.

The author has apparently achieved his major objectives. The manual can be recommended as an inexpensive source of student information and directions for typical, well-planned laboratory operations. The thirty-six experiments provide sufficient material and latitude for a traditional well-balanced one year organic course. The book should take its rightful place among the good semimicro organic laboratory manuals now available.

RALPH E. DUNBAR

North Dakota State College
Fargo, North Dakota

• LECTURE DEMONSTRATIONS FOR HIGH SCHOOL CHEMISTRY

Fred T. Weisbruch, **Head of Science Department, William Cullen McBride High School, St. Louis, Mo. Educational Publishers, Inc., St. Louis, 1951. 333 pp. 118 figs. 14 × 21.5 cm. $4.50.**

Fred Weisbruch's collection of lecture-table demonstrations comes as a timely aid to an art that needs revitalizing in high schools. Both the beginning teacher and the experienced demonstrator can gain valuable help from this little volume.

The demonstrations are arranged according to topics; line drawings are provided for unusual setups; appropriate precautions are included with hazardous experiments; and a goodly supply of audio-visual shocks is included in the form of bangs and flares.

It is unfortunate that the apparently high price for a lithoprinted book may limit its distribution. The clear text is unusually free from misprints, and the entire book attests to careful work in its preparation.

References to some of the articles written by inspiring demonstrators (Charles Stone, for example) are abundant. Most of the references are to This Journal. Unfortunately, use of many of these references requires publications that are now over 20 years old. It is doubtful that many chemistry teachers have this resource.

Some confusion exists in the name of PbO_2 which is called both lead peroxide and lead dioxide. Many of the qualitative experiments are those that the student should do for himself in the laboratory. One may question the value of lists of materials placed after groups of experiments.

This book will help any elementary chemistry teacher. Therefore it is highly recommended.

ELBERT C. WEAVER

Phillips Academy
Andover, Massachusetts

• INORGANIC CHEMISTRY, AN ADVANCED TEXTBOOK

Therald Moeller, **Associate Professor of Chemistry, University of Illinois. John Wiley & Sons, Inc., New York, 1952. ix + 966 pp. Illustrated. 15.5 × 23.5 cm. $10.**

Part I of this book includes a discussion of the theoretical background which is essential to the understanding of the factual material, which is covered in Part II. The presentation in Part I follows a logical sequence: atomic nuclei and their properties; the extranuclear structure of atoms and the periodic classification of the elements, and the properties dependent on the extranuclear structures; valency and the chemical bond; complex ions and coordination compounds; oxidation-reduction processes. Part I also includes a discussion of acids and bases and of nonaqueous solvents. In Part II, after the discussion of the inert gases and of hydrogen, the chemical elements are covered mainly in the reverse order of the Periodic Table : Groups VIIb, VIb, Vb, IVb, IIIb; followed by Groups I and II and the transition elements

The special appeal of this work to the teacher and the student of inorganic chemistry lies in the fact that an attempt has been made to gather into one place material which hitherto has been available only by reference to a variety of sources. The selection of this material has been thoughtfully planned and the result is a very teachable textbook, clearly written and with a minimum of mathematical treatment, up to date, comprehensive, with ample references to the original literature for those who would seek further enlightenment.

Professor Moeller is to be congratulated in supplying a much-needed aid to the instructor in advanced inorganic chemistry, whether at the senior or graduate level. The book should go far to dispel the often-heard but erroneous belief that inorganic chemistry has nothing left to explore, that everything of consequence has been done, and that the search for new things lies rather in the other branches of the science of chemistry. A careful reading of this book will reveal that inorganic chemistry is, in fact, a most fertile field of research and is concerned with many of the developments which claim the interest and attention of present-day scientists, technologists, and industrialists.

WALTER C. SCHUMB
MASSACHUSETTS INSTITUTE OF TECHNOLOGY
CAMBRIDGE, MASSACHUSETTS

● COMMUNICATION OF TECHNICAL INFORMATION

Robert M. Dederich. **Chemonomics, Inc., New York, 1952. 116 pp. 15.5 × 21 cm. $5.**

NOTHING except the promise to review this book for readers of THIS JOURNAL could have induced this reviewer to persevere to the end of it, doggedly turning page after page and reading every word. It took a long time, but here goes.

This book offers a supreme example of something in which one "can't see the forest for the trees," because the physical appearance of any page (all off-set from heavy typewriting, with ragged right margins) was so distracting that it was often difficult to sense the meaning of the text. The typographical errors thus embalmed were annoying, and the inevitable monotony of the crowded pages soon became most irritating.

Because any book on the communication of technical information might be eagerly seized by any scientist or technologist bent on improving his reports or other written work, intellectual honesty compels the flat statement that this little book, alas, will not be very helpful. It is simply baffling in the frank abandon with which the author blandly admits all that his book is not. It is, for example, ". . . not on report writing . . . simply about reporting." The chapter on writing is just about writing". . . not an attempt to provide a guide for punctuation or spelling or grammar; rather it is an attempt to show the relationship between writing and thinking." Further, ". . .the thinking comes more from . . .anthropology and psychology than from . . .literature."

The foreword, by an Assistant Professor at Columbia University (department not indicated; not in "American Men of Science"), says that the author is "uniquely fitted to write in this field" because of his academic training in English literature, rhetoric, and grammar, plus engineering school while in the Navy. Such a flaunting promise of excellence is best fulfilled by a text of which every page exemplifies "Q.E.D." It seems incredible that anyone actually trained in English could use such poor English in general, such a childish presentation in spots, and so many appalling sentences. It seems to have been left entirely without editing.

The text is freely interlarded with the jargon of "pedaguese" (*e. g.*, "area," "frame of reference," "pattern of activity," "postulated entity," "structuring"). Many pages seem to be merely a rehashing of notes taken in some course (probably one of these new-fangled offerings on "Communications" under Social Studies), in which the professor starts droning on a balmy September day, knowing that he has until Christmas to get his points across. The whole thing, in fact, might well have started as a "literary mosaic," like some of the term papers submitted in just such courses.

This book is literally incredible; that is, it must be seen to be believed. One consolation for disappointed persons who may have paid the unaccountable price of it is that for anyone seriously interested in the communication of technical information it is a treasury of "horrible examples" on which to practice editing and the correcting of both obvious and elusive errors of all kinds.

FLORENCE E. WALL
NEW YORK, NEW YORK

● THE COMMON AND SYSTEMATIC NOMENCLATURE OF THE SIMPLER ORGANIC COMPOUNDS

***Fred Semeniuk,* Professor of Pharmaceutical Chemistry, School of Pharmacy, University of North Carolina. North Carolina Pharmaceutical Research Foundation, Inc., Chapel Hill, 1952. vi + 55 pp. 21.5 × 28 cm. Paper covers. $1.25.**

THE number of known organic compounds, variously estimated around half a million, is increasing rapidly. This has brought about a strong demand for accurate names and a growing interest in nomenclature. Books on this subject are needed, and in fact two such have already appeared, Mitchell's in England and Gaede's in Holland. Unfortunately this American addition to the list is a distinct disappointment, being as likely to mislead students as to help them. It contains numerous errors, especially in the "I.U.C." names (there is no recognition of the fact that the IUC became the IUPAC, International Union of Pure and Applied Chemistry, in 1949, or of the decisions made in 1949 and 1951). The IUPAC rule that only one kind of function will be expressed by the ending of the name is disregarded; so also is the rule that the carbon atom having the free valency, in an aliphatic hydrocarbon radical is numbered one (hence such radical names as 2-propyl for isopropyl are wrong). It is not true that "oxo" is interchangeable with "oxy." Instances of wrong correlation of structure with name are: —O—O—, dioxo; —N=N—, azo; O=C=C=O, oxalyl. Some of the structures, as of azides, ozonides, calcium carbonate, are not modern.

There are three or four pages of explanation; the rest of the material is in tabular form.

AUSTIN M. PATTERSON
XENIA, OHIO

● CHEMISTRY OF CARBON COMPOUNDS. VOLUME I, PART B: ALIPHATIC COMPOUNDS

Edited by *E. H. Rodd.* Elsevier Publishing Co., Houston, Texas, 1952. xvi + 684 (779–1462) pp. 23 figs. 15.5 × 23.5 cm. Subscription price, $15; single copy, $17.50.

"CHEMISTRY of Carbon Compounds" will constitute a five-volume comprehensive treatise on organic chemistry when completed. Volume I covers a general introduction and aliphatic compounds, while Volumes II to IV will consider alicyclic, aromatic, and heterocyclic compounds, with Volume V containing miscellaneous material and general indexes. To date Volume I has been published in two parts, IA (pages 1–778, Introduction and Chapters I–XI (1951)) and IB (pages 779–1462, Chapters XII–XXIII). Future volumes are scheduled to appear at approximately ten-month intervals. The entire series is under the general editorship of Dr. E. H. Rodd, formerly scientific liaison officer between the Dyestuffs Division of I.C.I. and the British Universities. Individual chapters to the volumes have been contributed by one or more distinguished British chemists. There are 23 contributors to Volume IA and 13 to Volume IB, five of the group having contributed to both volumes. Dr. Rodd is assisted in his editorship by an advisory Board including such eminent British scientists as Professors R. Robinson, J. W. Cook, R. D. Haworth, I. Heilbron, E. L. Hirst, and A. R. Todd.

The intention of the present reference series is to bridge the gap between such encyclopedias as Beilstein's "Handbuch" and ordinary single volume organic texts. The outlines of the present

undertaking have been patterned after Richter's "The Chemistry of the Carbon Compounds," the third English edition of which became available between 1934 and 1947. The Richter classification has been retained in the present series in view of the editor's feelings that "any attempt to change it might well create more difficulties than it removed," and that organic chemists already have a working familiarity with it. In one sense then, the present series is admittedly and intentionally another revision of "Richter." In a broader sense, however, this evaluation is inadequate in that the treatment of most topics is not only expanded and brought up to date but also presented in a more unified manner. The subtle differences permit the present undertaking to stand on its own merit, independent of its illustrious predecessor.

One important criterion of adequacy in a reference series of the present sort is the timeliness of its documentation, and your reviewer was particularly interested in this question regarding Volume IB. The volume contains over 5300 references. Of these, approximately 16 per cent appear in the literature prior to 1900, 32 per cent between 1900 and 1930, 23 per cent between 1930 and 1940, and 29 per cent between 1940 and 1952. Literature selection has thus been heavily weighted in favor of recent material, and the volume would seem quite acceptable as a guide to current information on the topics considered. As further insurance of adequacy as a reference text, the volume has a 70-page subject index containing around 7500 entries. Almost 200 different periodicals are listed as source material for the subject matter of the present volume, and extensive reference to patent literature is included.

A number of other distinctive features round out the excellence of the present volume. Page references to past and future volumes are included in the text when pertinent. Frequent reference is made to the historical aspects of the topics under discussion, and numerous references are given to more extensive review articles. Considerable emphasis is given to physical properties of the compounds discussed, as well as to physical properties of their numerous derivatives. This feature is occasionally handled in extensive tables, but more usually merely as part of the text. Broad consideration is given to biological and biochemical aspects of the topics treated, and the volume contains up-to-date treatment of carbohydrates, proteins, and enzymes in its latter chapters. Occasional reference to syntheses of isotopically labeled substances is made, but in general there is little emphasis in this direction. The over-all approach to the subject matter is factual rather than theoretical, the order of presentation being preparations, then reactions, with physical property data interspersed where pertinent. Theoretical material is purposely included only incidentally, but occasional reference is made to both the theoretical literature and to a more unified treatment of theoretical organic chemistry in Volume IA. Analytical organic chemistry is almost entirely considered from the viewpoint of modern micromethods. Despite the large number of individuals contributing to the volume, there is an impressive and gratifying stylistic homogeneity about the book, and one fails to receive the impression of helter-skelter so often conveyed in works written under contributory authorship.

The book is not without some objections, but these are in the main quite trivial. Although unified in all other ways, Volumes IA and IB have separate indexes, and no author index is included, undesirable features which will presumably be rectified in Volume V. It is also hoped that a formula index will be included in Volume V, both because of the general magnitude and complexity of the work, and because British nomenclature occasionally differs slightly from our own. The editor might have sanctioned a more extensive use of equations and formulas, although the reasons for their omission are obvious. Similarly, the occasional reference to reactions by discoverer's name only might sometimes prove frustrating. Regarding both textual statements and derivatives listed, references are often omitted, so that the volume suffers somewhat as an immediate bridge to the original literature. This criticism is not as serious as might be, however, since it applies mainly to the earlier literature. The frequent use of "*loc. cit.*" is annoying in that it necessitates more extensive perusal to find the reference in question. Reference style is not strictly uniform throughout the book and nomenclature, especially in the carbohydrate chapters, is occasionally antiquated or incorrect. In Chapter XVIII no attention is paid to uniform configurational designation, *d*-, *D*-, and (+) −, for example, being used indiscriminately and ambiguously.

In general, however, the volume is remarkably free from both typographical and, apparently, factual errors. A sheet of "Addenda and Corrigenda" to Volume IA is incuded with IB, a practice which will presumably be continued in future volumes. It is interesting that this sheet contained only 14 additions or corrections to Volume IA. The number of such errors in the present volume does not appear much greater. In fields where your reviewer had some familiarity, it appeared that the incidence of important omissions was not particularly high.

If future volumes in this series maintain the high standards to be found in the present volume, the series is certain to find a key position on the bookshelves of most practicing organic chemists. From the viewpoint of quick, accessible, up-to-date information at moderate cost, the present undertaking looks highly promising.

WILLIAM A. BONNER

Oak Ridge, Tennessee

● AN INTRODUCTION TO THE CHEMISTRY OF THE HYDRIDES

Dallas T. Hurd, Research Associate, General Electric Research Laboratory. John Wiley & Sons, Inc., New York, 1952. x + 231 pp. 4 figs. 15 × 23 cm. $5.50.

This book conforms well to its title, for it may be read with some profit by chemists who know very little about inorganic hydrogen compounds, and it emphasizes substances having hydridic reaction tendencies rather than any appreciable protic acid character—substances which must become far more familiar to the chemical public if the most neglected parts of the chemistry of hydrogen are to progress as well as they deserve. The author presents some of the least known aspects of the subject almost as fully as knowledge permits, while omitting much of what is known or understood about topics which are surveyed by advanced books. This principle of selection correlates fairly well with a tendency to treat most accurately the subjects most closely related to the author's own very significant research studies—although his effort to cover a far broader field is not a complete failure.

The presentation is fairly clear and at some points even careful; and the novice will be able to extract some useful hints as to what should be studied further in order to arrive at a good understanding, even though the literature references are very meager. The general pattern of organization would be good if it were well filled out: first a classification of types of hydrogen compounds; then in Chapter 2 there is a quick survey of chemical bonding principles, with especial reference to hydrogen compounds; then the descriptive chapters are developed in the periodic-system order, with explanatory chapters interspersed for purposes of integration of ideas and stating generalizations.

Experts who are well informed on the chemistry of inorganic hydrogen compounds inevitably will dislike this book for its frequent errors of fact or meaning, for the superficiality of its attempt at integration of similar topics, and for its lightness of contact with subjects deserving better discussion. They will also note with dismay an occasional tendency to generate alleged facts from one-variable theories. The most glaring example of this is the wide application of the author's evident belief that hydrides of heavier elements in a periodic group behave chemically according to the electronegativity rules observed in right-to-left series-trends. Thus on page 153 one reads that Se and Te are good electron-donors (which they are not) and that their hydrides have practically no tendency to form hydronium ions in water. Actually, K_1 for H_2S is 1.15×10^{-7}; K_1 for H_2Se is 1.7×10^{-4}; and K_1 for H_2Te is 2.3×10^{-3}—a trend of increasing acidity opposite to the decrease of ionic character of the bonding, but quite

typical of the hydrogen compounds of all groups in which protic-acid character can be detected by any means. The author apparently believes that a generalization is useful if it is true half the time for he often puts half-truths forward as "generally" true, and then says one should not trust them very far. Inconsistencies of detail also appear: thus on page 103 it is stated that it is not known for sure whether electrolysis is applicable to the preparation of hydrides of silicon, germanium, or carbon, but on page 113 it is said that low yields of germane may be obtained by that method. In sum, it would seem that the subject is too large for the space assigned, and that the author has tried to extend himself beyond what he has had time to understand thoroughly.

ANTON B. BURG

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● GMELINS HANDBUCH DER ANORGANISCHEN CHEMIE. SYSTEM 3: SAUERSTOFF

Eighth edition. Verlag Chemie, GMBH, Weinheim, Germany, 1952. 218 (83–300) pp. 32 figs. 17.5 × 25.5 cm. $15.48.

THIS bulletin deals with the occurrence of oxygen, ozone, and water; the technical preparation of oxygen, ozone, and hydrogen peroxide; and the purification of water. It is a good source of material on the geochemistry of oxygen, ozone, and water, and is fairly well up to date in such matters as isotope distribution and the cosmic distribution of both oxygen and water. Little, if any, of the literature within the last five years seems to have been covered, but this may perhaps be expected in a handbook so extensive as this one. Every individual section of such an extensive handbook must be considered from the standpoint of its significance for specialists in a particular field. This section satisfies such a requirement.

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● GMELINS HANDBUCH DER ANORGANISCHEN CHEMIE. SYSTEM 17: ARSENIK

Eighth edition. Verlag Chemie, GMBH, Weinheim, Germany, 1952. xv + 475 pp. 20 figs. 17.5 × 25.5 cm. $33.33.

IN KEEPING with other units of this work which have appeared recently, the present volume represents a marked improvement over the corresponding portion of the seventh edition of the Gmelin-Kraut Handbuch, Volume III, Section 2, edited by C. Friedheim and published in Heidelberg by C. Winter in 1908. The revision has been thorough, both in the elimination of much superfluous material, and in the inclusion of considerable more recent information. The literature coverage extends through December, 1949. The new format of the eighth edition, with its greatly improved typography, makes for increased ease of reading; and the elimination of much of the "cookbook" style in the passages dealing with the preparative aspects of the subject is also a welcome change.

The subject matter includes a brief historical introduction (9 pages), followed by an extensive treatment of the occurrence of the element and its compounds (74 pages), and of their uses (4 pages). The formation and preparation of the element in its several forms (12 pages) is followed by a thorough review of the physical and chemical properties of the element (87 pages) and of the physiological effects of both elementary arsenic and its compounds (8 pages). A detailed account of the compounds of arsenic with hydrogen, oxygen, nitrogen, and the halogens; with sulfur, selenium, and tellurium; and with boron, carbon, silicon, and phosphorus completes the volume (281 pages).

The general style and thoroughness of treatment are maintained at the high level which has come to be expected in this standard comprehensive work of reference. As a single example of this thoroughness, the section dealing with the physical properties of arsenic may be pointed out. It begins with a few general remarks, then turns to the nuclear characteristics of the various isotopes and the transformations induced therein by bombardment with different projectile particles. The outer atom is next described in detail, followed by a consideration of the arsenic molecule. In succession the crystallographic, mechanical, thermal, optical, magnetic, electrical, and electrochemical properties are covered, each of these topics being duly subdivided into their pertinent classifications.

The editorial staff of more than a score of men and women who have been engaged upon the revision of this volume have added one more block to the monumental structure that constitutes the eighth edition of the Gmelin "Handbuch," which when completed will doubtless represent the world's most reliable and authoritative survey of the entire field of inorganic chemistry.

WALTER C. SCHUMB

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● GMELINS HANDBUCH DER ANORGANISCHEN CHEMIE. SYSTEM 27: MAGNESIUM

Teil A, Lieferung 4: Legierungen von Magnesium mit Zink bis Rhenium. Eighth edition. Verlag Chemie, GMBH, Weinheim, Germany, 1952. xiv + 336 (493–818) pp. 86 figs. 17.5 × 25.5 cm. $24.

THE eighth edition of Gmelin's famous handbook is similar in style and arrangement to the earlier editions, and little description of it need be given. As with the earlier editions, the book is being published in small units, each of which is published as soon as it is ready. This enables the reader to get information which is fairly well up to date. For example, the book being reviewed here covers the pertinent literature up to the end of 1949. Unfortunately, the different portions of the work do not always cover logical divisions of the subject matter. This volume discusses the alloys of magnesium with the metals in periodic groups IIB, III (except aluminum), IV (except silicon), VB, VIB, VIIB, VIII, and IB, and the surface treatment of magnesium and its alloys. The alloys of magnesium with silicon, and with the metals of Groups IA, IIA, and VA were included in an earlier issue and the alloys of magnesium with aluminum will be included in the volume on aluminum. Magnesium compounds will be discussed in Part B of the magnesium section.

This book is indispensable for those who are working with the alloys of magnesium, for it contains a complete bibliography on the subject as well as brief descriptions of much of the work in the field. Many phase rule diagrams are included. While this volume of the series will have little appeal to the general reader, every chemical library should have a complete set of "Gmelin," for it is by far the most complete reference book of inorganic chemistry.

JOHN C. BAILAR, JR.

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URBANA, ILLINOIS

● STATISTICAL TABLES AND FORMULAS

***A. Hald.* John Wiley & Sons, Inc., New York, 1952. 97 pp. 19 tables. 22 × 28 cm. $2.50.**

THIS pamphlet is composed of two parts. The first part contains fundamental formulas and examples of the uses of the various tables that follow in part two. Frequent reference is made to the author's text, "Statistical Theory with Engineering Applications."

Among the standard tables to be found in part two are tables of the normal density function, the normal distribution function, probits of the normal distribution, percentiles of the t, χ^2, χ^2/d.f., and F distributions and confidence limits for the parameter of the

binomial distribution. The above tables contain 21 percentiles of t and χ^2, and 17 percentiles of F, constituting an extension of previously published tables. By including the 97.5, 99.5, and 99.95 percentiles for t and F, the author has, in effect, provided critical values for "two-tailed" tests at the 5 per cent, 1 per cent, and 0.1 per cent levels, respectively.

There are, in addition, tables of the distribution of the range, the one-sided truncated normal distribution, and the one-sided censored normal distribution. The range table includes 21 percentiles and the first two moments for sample sizes from 2 to 20.

The remaining tables are devoted to the "arc sine" transformation, logarithms of factorials and binomial coefficients, squares, square roots, reciprocals, logarithms, and random sampling numbers.

EDGAR P. KING

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● THE PETROLEUM CHEMICALS INDUSTRY

Richard Frank Goldstein. **John Wiley & Sons, Inc., New York, 1950. xiii + 449 pp. 41 figs. 92 tables. 5 charts. 16 × 25 cm. $8.50.**

Petroleum is one of three large-scale sources of raw materials for the chemical industries. Hence it is not surprising that so many processes have been devised to manufacture the finished products demanded by our level of civilization. What is surprising is that this book, written by an Englishman describing in the main American industries and processes, happens to be the only book of its kind. (This excludes, of course, the compilation of Ellis.) This reviewer can certainly concur with Sir Robert Robertson, who wrote the Foreword that "this work deserves to be termed monumental..." For within 20 chapters an amazingly large fund of information is found about chemicals and chemical reactions. Practically all of the materials are those taken from publications since 1940.

Abbreviations of journals and companies inaugurate the text; an introduction giving a brief review of nomenclature and thermodynamic symbols and data follow. The first chapter, Sources of Petroleum Hydrocarbons, includes composition, tables of physical properties, and process of refining.

The next ten chapters are mainly concerned with the synthesis and reactions of aliphatic chemicals from petroleum. Heavy emphasis is placed upon the role of the unsaturates. Tables of properties, charts of reactions, short flow diagrams, techniques of purifications, and uses to which these chemicals are put are all emphasized. Other topics included in the text are: naphthenes, aromatics, acetylenes, aldehydes, ketones, acids and acid derivatives, ethylene oxide, nitriles, and amines. Chapter 20 deals with by-products of interest to other industries. These include nitrogen, oxygen, and sulfur compounds.

Appendix I contains five reaction chart summaries, one of which shows how coal or fermentation processes can be used as sources of aliphatic chemicals. Appendix II has eight tables of statistics on petroleum production and comsumption. Appendix III contains data and statistics on synthetic organic chemicals in the U.S.A. for the years 1921—45. Four indexes complete the book. A large number of folded charts are contained which are comprehensive, and offer many lesser known reactions. These charts help make the book more valuable as a reference.

By necessity, the elementary organic texts cannot cover petroleum chemistry adequately. Yet every instructor does go beyond the text he assigns in discussing what useful chemicals can be made from hydrocarbons. In Dr. Goldstein's book, essentially for the first time, assignments can be made to find answers for specific questions as: Why was thiophene sold by a petroleum company? What is the synthetic process for the manufacture of glycerol? What are some of the polymerization products of acetylene? What are naphthenes?

Naturally, no book of about 400 pages of text is absolutely complete, and certainly much has been accomplished since the text was printed. However, practically all immediate information that the organic instructor needs can easily be found in this work. Every school library should have a reference copy. Where petroleum chemistry courses are given, this may be the very text sought.

ARTHUR FURST

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● STRUCTURAL ASPECTS OF CELL PHYSIOLOGY

Symposia of the Society for Experimental Biology, Number VI. Published for the Company of Biologists on behalf of the Society for Experimental Biology. Academic Press, Inc., New York, 1952. vi + 357 pp. Illustrated. 16 × 25.5 cm. $7.50.

The morphological and the physiological approach to biological problems have developed largely independent. Attempts to bring both lines closer together are evidently of great importance and one of the prerequisites for the ultimate goal, a full understanding of the life process. The present book is a useful summary of what is being done at present to achieve such a synthesis in one segment of the biological sciences, that of cellular life. It comprises 21 papers presented at Bristol in July, 1951, 15 originating from British, two each from American and Swiss, and one each from Swedish and Belgian laboratories.

The contributions appear to be grouped roughly around three main themes. The first eight or nine papers deal largely with various aspects of membrane properties. Danielli discusses the factors important in cell permeability and secretion, stressing the importance of the theory of activated diffusion and pointing out that phosphatases, in secretion, may be of significance primarily as contractile proteins. Katz reviews the properties of the nerve membrane and its relation to the conduction of impulses. Runnström has an extensive chapter on cell surface in relation to fertilization, based almost entirely on experiments done with sea urchin eggs. The following two papers by Swann and Mitchison, respectively, are closely connected, presenting a theory relating mitosis and cleavage. Goldacre studies and illustrates by means of very clear microphotographs the action of anesthetics on amebas, while Kitching's paper deals with the contractile vacuoles of protozoa, discussing such features as the mechanisms of systole and diastole and the question of osmoregulation. Coons, in a very brief chapter, states that intravenously injected antigens find their way in a surprisingly short time into the cytoplasm and even the nuclei of a variety of tissues.

The next series of seven papers is grouped around the theme of growth and development. Brachet discusses the respective roles of nucleus and cytoplasm in synthesis and morphogenesis, developing the interesting view that the nucleus participates in the coupling between oxidations and phosphorylations. Sanders studies the mechanism of virus reproduction within cells. He brings out the point that the virus parts responsible for the initiation of multiplication are smaller than the infectious units, and that hence the conclusions drawn by geneticists from findings on completed virus particles may be misleading. Pontecorvo presents a genetical analysis of cell organization, while Baltzer reviews the behavior of nuclei and cytoplasm in amphibian interspecific crosses, a field to which he has contributed so much. Callan draws attention to the potentialities offered by amphibian oocyte nuclei for experimental research. Hughes points out that inhibitors of mitosis have so far been used primarily for practical purposes rather than fundamental analysis for which they might be quite important if applied to suitable forms. Walker and Yates studied the ultraviolet absorption of living nuclei during growth and development, a procedure destined to supplement the purely biochemical approach.

While the first two series of papers dealt largely, though not exclusively, with animal cells, the last six contributions are devoted to plant cells. A very instructive picture of the structure of the yeast cell is given by Lindengren, while that of the root meristem of *Vicia faba* is discussed by Chagen. Manton's interesting account of the fine structure of plant cilia is illustrated

by a truly remarkable series of beautiful electronmicrographs. The next two chapters deal with growth problems; Frey-Wyssling discusses that of the plant cell walls, while Brown, Reith, and Robinson study the growth of plant cells mainly in isolated fragments but also in intact organs. The final chapter by Preston discusses the biological units of cellulose structure derived from a study of the vesicles of *Valonia*.

All chapters are concisely written by experts in their chosen fields and most of them are illustrated either by very clear diagrammatic text figures or by microphotographs and electronmicrographs on plates, the latter being among the best this reviewer has seen. Each paper has its own bibliography. The volume will appeal primarily to biologists, but will be of great value also to those biochemists and biophysicists who wish to correlate their findings with structural elements. The only regrettable feature of the book is that it contains neither an author nor a subject index.

THEODOR VON BRAND

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● CATALOGUE OF PRINTED WORKS BY AND MEMORABILIA OF ANTOINE LAURENT LAVOISIER: 1743–1794

Printed in limited edition of 1000 copies for the Grolier Club by the George Grady Press and privately distributed. 70 pp. 14 × 22 cm. Paper bound. $2.

NOWHERE in the world is there a more complete private collection of the works of Antoine Lavoisier than that of Mr. Denis I. Duveen. Through Mr. Duveen's generosity, the Grolier Club was enabled to exhibit this collection to the public in February and March, 1952.

From the earliest papers on meteorology through his most significant scientific contributions, and not omitting his many contributions in the fields of economics, finance, agriculture, politics, sociology, education, and reform, the collection reveals again what we often forget—the amazing versatility and sound judgment in all fields exhibited by the "Father of Modern Chemistry."

The papers on combustion start with the early work of Lavoisier, Macquer, and Cadet on heating the diamond, and cover all of the important contributions at this significant period in Lavoisier's thinking through the high point of the Revolution reached in his "Mémoire sur la nature du principe qui se combine avec les métaux pendant leur calcination, et qui en augmente le poids." This paper, a classic in the history of science, was published first in 1775 and later, in revised form taking into account Priestley's discovery of dephlogisticated air, in 1778.

Unique in the collection is a first edition of "Traité élémentaire de chemie," of which only three copies are known. Only recently has it become apparent that the edition commonly regarded as the first was actually the second.

Scientists know Lavoisier as a scientist. His writings and activities in other fields, however, while overshadowed by the revolution he achieved in science, entitle him to standing as a contemporary thinker of distinction in all public fields. The collection includes some 20 papers in fields other than science. Among them is his "Reflexions sur l'instruction publique" on the reorganization of the educational system, which document shows extraordinary insight into the problems of education in France. Indeed, Talleyrand sent him his own draft on the subject, begging criticism. Again, his "De la richesse territoriale de la France" is a contribution of highest order in the political economy of France. Many other papers in the exhibit on agriculture, hospitals, and his work in the Provincial Assembly of Orleans attest to his broad talents. In 1792 he was offered the post of Ministre des Contributions Publiques, which in view of the troubled times, he promptly declined. That a man with such liberal views should become a victim of the contemporary hysteria which demanded liberation or heads is still an enigma.

The collection includes a number of personal items, among them a music manuscript of 48 pages in Lavoisier's hand; a passport issued to Lavoisier in 1792 granting him permission to go to Blois; a list, including Lavoisier's name (spelled Savoisier), of those condemned to death by the Tribunal; and a number of personal letters. Only one authentic portrait of Lavoisier is known. Painted by the famous Jacques-Louis David, it hangs in the Rockefeller Institute for Medical Research. Many other pictures, however, have been made, either as indirect copies or as imaginative conceptions. Some 14 of them are included in the collection, together with several bronze medals and the commemorative French Stamp of 1943 marking the 200th anniversary of Lavoisier's birth.

The generosity of Mr. Duveen in making this superb collection available, and the courtesy of the Grolier Club in presenting it for public exhibition and preparing the Catalogue is deeply appreciated by all those interested in the history of science. We need to be reminded again and again of the genius of this man whose last days were such a tragic anticlimax.

SIDNEY J. FRENCH

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● OPTICKS

***Sir Isaac Newton.* Based on the fourth edition, London 1730. Foreword by *Albert Einstein.* Introduction by *Sir Edmond Whittaker.* Preface by *I. Bernard Cohen.* Dover Publications, Inc., New York, 1952. cxv + 406 pp. 8 photographs. 56 figs. 6 tables, 11.5 × 17.5 cm. $1.90, paper bound; $3.95, cloth bound.**

THIS edition of Newton's "Opticks," like the edition published by Whittlesey House, McGraw-Hill Company in 1931, is based on the fourth edition of 1730, the edition published three years after Newton's death, which had, however, been corrected and made ready for publication by Newton himself. It is dedicated to Marjorie Hope Nicolson, "who uncovered the influence of the Opticks on 18th-century imagination." The foreword by Professor Einstein, two pages, and the introduction by Professor E. T. Whittaker, both written for the 1931 edition, are included in this volume. These inclusions are preceded by the long preface, 40 pages, by Professor I. Bernard Cohen, and are followed by a detailed summary of the contents of the book proper, except for the last fifteen queries. This synopsis takes up 32 pages.

The excellent preface by I. Bernard Cohen makes this edition of the "Opticks" a most valuable addition to the library of any physicist or chemist interested in the history of science in the late seventeenth century. Professor Cohen places the "Opticks" in its proper setting not only with respect to the development of the experimental and theoretical aspects of light in this period, but also with respect to the scientific ideas expressed by other writers of the time. More important than this, however, is the lengthy and discriminating comparison of the two great classics that came from the work of the mind and the hands of this intellectual giant, the "Principia" and the "Opticks". "While the 'Principia' seemed the terminal point of an ancient line of inquiry, the 'Opticks,' with its newly discovered phenomena concerning colors and diffraction, clearly marked the beginning of a new direction in physical inquiry."

As Professor Cohen remarks, the "Opticks" is "one of the most readable of all the great books in the history of physical science." Of particular interest to the chemist are certain of the Queries. Most of the experimental work on light reported in the book was done in the early years of Newton's life in his laboratory in Cambridge. It is often forgotten that Newton devoted a goodly portion of his time for more than 25 years to investigations in chemistry, mainly with metals, fire, and flames, and that this experimental work parallels the investigations on prisms and lenses. Robert Hooke in London pursued similar investigations. The ideas and personalities of these two greatest geniuses of seventeenth-century England clashed. The first edition of the "Opticks"

appeared in 1704, but it is highly probable that it was completed by 1692. Newton deliberately delayed publication until after the death of Hooke in 1703.

The copy of this edition sent to the reviewer was the paperbound student's edition. The paper and print are excellent, the errors are few; but because of the thickness of the volume and compactness of the binding it is difficult to handle.

CLARA DE MILT

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• MALEIC ANHYDRIDE DERIVATIVES

***Lawrence H. Flett* and *W. Howlett Gardner*, New Products Division, National Aniline Division, Allied Chemical and Dye Corporation. John Wiley & Sons, Inc., New York, 1952. x + 269 pp. 15 × 23 cm. $6.50.**

THIS book offers a condensed presentation of 116 different types of chemical reactions involving additions to maleic anhydride in most instances, although some cases dealing with maleic acid, fumaric acid, their esters and amides or other unsaturated derivatives are included. For clarity and conciseness each type reaction is confined to two open pages. The name of the reactant and structural equation of the reaction, a brief experimental procedure as taken from one major reference and supplementary references and patent numbers through 1950 occupy the right page. On the opposite page there is a further appraisal of the applicability of the method, a listing of the physical properties, as well as the present and some suggested uses of the product.

The reactions are organized in eight groupings presenting the addition of (1) hydrocarbons, (2) halogen and halogen compounds, (3) hydrogen, (4) metallic compounds including metallo compounds, Grignard reagents, and Friedel-Crafts reactions, (5) a wide variety of compounds containing nitrogen, (6) compounds containing oxygen, (7) sulfur compounds, (8) the effects of light and heat alone.

The authors have organized much scattered material into a useful and compact reference treatment. In doing so they have drawn upon their wide industrial experience in making choices and in perspective. Certainly the organic research worker and the industrial chemist will find a chance perusal worth while and in many instances creative suggestions will result.

HAROLD A. IDDLES

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• A GUIDE TO THE HISTORY OF SCIENCE

***George Sarton*, Harvard University. Chronica Botanica Co., Waltham Mass. 1952. xvii + 316 pp. 15.5 × 23.5 cm. $7.50.**

THIS work by the world's leading historian of science can be considered to be an outgrowth of the two journals in this field, *Isis* and *Osiris*, which Sarton founded and has long edited. For this reason he uses as a short title for this book the word *Horus*, the name of the son of Isis and Osiris, and he intends that in future the guide should be referred to by this name.

The book is divided into two parts. The first, entitled "Science and Tradition," consists of three essays on the importance of and need for teaching the history of science as a part of the cultural heritage of western civilization. Sarton is no longer as optimistic as he once was that courses in this subject, or in the history of the specialized sciences, will soon be established in most of our higher educational institutions. He is none the less eloquent and convincing in showing the necessity for such courses, not only for the scientist but for all educated men. Of course, the scientist is in particular need of such courses, so that he may understand to the full the forces that have made his science, and to a considerable degree himself, what they are. The chemist is no exception, and the narrow specialization of most of our graduate schools is increasingly being recognized. The study of the history of chemistry could do much to counteract this trend. No small reason for the lack of interest in the historical phases of the science shown by our graduate students lies in the indifference with which the subject is treated by their professors, themselves a product of the same indifference. The natural interest felt by many younger chemists is quickly stifled in such an atmosphere. It is to be hoped that these essays will be read and considered thoughtfully by the heads of chemistry departments throughout the country.

The second part of the book is designed chiefly for the specialist in the history of science. It is an elaborate bibliography of all the literature known to Sarton on the various phases of the history of science. Following the listing of general reference works, such as gazeteers and encyclopedias, book titles are arranged under the headings of special countries or cultural groups, special sciences, and journals and publications of special groups. This means that titles pertaining to any one science, such as chemistry, must be sought in several places, but the bibliography is not so long as to make this task unduly difficult. Most of the entries contain bibliographic information only, but sometimes the author includes brief critical notes to characterize the books more fully. Although this section will be useful most often to specialists, almost any scientist may at times find it helpful to consult. The book is a unique work of reference which will long retain its value.

HENRY M. LEICESTER

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SAN FRANCISCO, CALIFORNIA

• DICTIONARY OF CIVIL DEFENSE

Edited by *Carlton Wallace*, Associate Fellow, Institute of Civil Defense. Philosophical Library, Inc., New York, 1952. 160 pp. 16 figs. 13 × 19 cm. $2.75.

THIS book covers about 400 definitions of words related to Civil Defense. It was originally published in Great Britain and is of interest as an indication of the type of organization now existing in a country which knew enemy attack and how to combat it on the home front.

Beyond satisfying this interest the book is of little value. One difficulty with it arises from its origin in Great Britain whose Civil Defense organization is so greatly at variance with its existing counterpart in the United States. Over one-third of the book is devoted to first-aid which is better presented in our own Red Cross handbook. There are certain technical errors which can hardly be attributed to language difficulty. The description of a Geiger counter fits more aptly an ion chamber and a quartz fiber electroscope is scarcely a Geiger counter as it is called. The recommended maximum permissible dosages of radiation do not agree with those recommended in the United States.

Although glossaries are needed in the field of Civil Defense they should accompany handbooks or manuals which are restricted to individual phases of the effort as protection in the event of enemy attack. The problem of Civil Defense is too complex to be successfully covered in such a dictionary as this.

PHILIP GILL

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• LINEAR COMPUTATIONS

***Paul S. Dwyer*, Professor of Mathematics, University of Michigan. John Wiley & Sons, Inc., New York, 1951. xi + 344 pp. 15.5 × 23.5 cm. $6.50.**

THIS is a textbook on calculating techniques for obtaining numerical solutions of sets of simultaneous linear equations with the aid of a desk calculator. Methods based only on elementary high-school algebra are described in detail in the first few chapters. Then the more powerful methods involving determinants

and matrices, and including the calculation of the adjoint, inverse, characteristic equation, and characteristic vectors are developed in later chapters. Specific applications of these techniques to statistical problems are treated in Chapter 18, and nonlinear problems are discussed briefly in the concluding chapter. All methods are well illustrated by examples, and there is a list of references and exercises at the end of each chapter.

A short introductory chapter discusses certain features common to most digital computing machines—the setting mechanism, revolutions register, and product register—and the operations of addition, subtraction, multiplication, division, and square root. The second chapter discusses approximate-error numbers, range numbers, "significant numbers," and incomplete numbers, and the manner in which errors combine under the rational operations and square root.

In Chapter 3 it is urged that the computational design be so devised that approximate operations (division or square root) be deferred as long as possible. Certain operations such as $(ab - cd)/e$, or $\sqrt{ab \pm cd}$, can be combined into operational units on a machine in such a way that only the final result needs to be copied or read from the machine. Error terms for each operational unit can be considered independently.

Chapters 4 and 5 are concerned with the solution of simultaneous equations by pivotal condensation methods in which certain second-order determinants are successively evaluated by multiplication and subtraction and serve to eliminate successively the variables $x_1, x_2, \ldots x_n$.

An exact division is included in the determinantal method of Chapter 5, where for $i > h$, $j > h$ the minor $d_{ij\cdot(h)}$ that includes rows $1, 2, \ldots, h, i$, and columns $1, 2, \ldots, h, j$ of the augmented coefficient matrix (a_{ij}) is computed by the formula

$$d_{ij\cdot(h)} = [d_{ij\cdot(h-1)}\, d_h - d_{ih\cdot(h-1)}\, d_{hj\cdot(h-1)}]/d_{h-1}$$

if $d_h = d_{hh\cdot(h-1)}$ denotes the principal minor from the first h rows and columns. In the abbreviated method the quantities $d_{ij\cdot(1)}$, $d_{ij\cdot(2)}, \ldots$, are computed successively, but the only recorded entries are the $n(n+1)$ quantities $d_{ih\cdot(h-1)}$ and $d_{hj\cdot(h-1)}$, for $h = 1, \ldots, n$; $i = h, \ldots, n$; and $j = h+1, \ldots, n+1$. In the back solution the unknowns $x_n, x_{n-1}, \ldots, x_h, \ldots, x_1$ are found successively in single operational units from the equations

$$\sum_{d=h}^{n} d_{nj\cdot(h-1)}\, x_j = d_{h,\ n+1\cdot(h-1)}$$

The use of row sum checks and symmetry where possible is discussed and illustrated.

Methods which reduce the elimination steps in the forward solution to operational rather than recording units are discussed and illustrated in Chapter 6. These include the methods of row division, of diagonal division, of single division; and abbreviated forms of these, such as the Gauss-Doolittle method for solving symmetric simultaneous equations, its generalization to nonsymmetric problems, and the modified square root method. Later (Chapter 13) it is shown that the last two methods are equivalent to the factoring of the given coefficient matrix a into the forms gb or $s's$, where g', b, and s are upper triangular matrices with diagonal entries G, 1 and $\sqrt{G}$ respectively.

In Chapters 7 and 8 certain algebraic relations between the various recorded numbers in various methods and in associated problems are studied. Then in Chapters 9 and 10 these quantities are identified as ratios of certain determinants. Here also the evaluation of a determinant whose entries are approximate-error numbers is discussed. Chapter 11 discusses the evaluation of the linear forms that arise in the solution of simultaneous linear equations.

The basic laws of matrices are set forth and illustrated in Chapter 12 as a background for the matrix methods developed in the next five chapters. The inverse of a matrix is then computed by various methods in Chapter 13. If a matrix a has elements a_{ij} and determinant Δ, and if A_{ij} is the cofactor of a_{ji} in Δ, then $A = (A_{ij})$ is called the *adjoint* of a, and its transpose A' (the untransposed cofactor matrix) is called the *adjugate* of a. The inverse matrix is A/Δ, if $\Delta \pm 0$. The underlying principle for finding the inverse by pivotal methods is the factorization of the matrix a into two triangular matrices. Chapter 14 describes a double bordering method and a method of Waugh and Dwyer for finding the adjoint.

Chapter 15 describes the methods of Bingham, Frame, and others for obtaining the characteristic equation $F(\lambda) \equiv |\lambda I - A| = 0$ and the adjoint $C(\lambda)$ of the matrix $\lambda I - A$. If constants c_k and matrices a_k are defined recursively by the equations

$$c_0 = 1,\ a_0 = I,\ c_k = -(1/k)\ tr\ a\ a_{k-1},\ a_k = a\ a_{k-1} + c_k I$$

then

$$F(\lambda) = \Sigma c_k \lambda^{n-k} \text{ and } C(\lambda) = \Sigma a_k \lambda^{n-k-1}$$

Characteristic vectors or modal columns for a simple root λ_k are nonvanishing columns of $C(\lambda_k)$. The so-called Frame method was discovered independently by Faddeev-Sominskii [FADDEEVA, V. N., "Computational Methods of Linear Algebra," (Russian), Gosudarstv. Izdat. Tehn.-Teoret. Lit., Moscow, Leningrad, 1950], and Fettis [*Quart. Applied Math.*, 8, 206–12 (1950)].

The so-called extension, or enlargement, or escalator methods discussed in Chapter 16 are based on formulas for computing the inverse of a matrix in terms of the inverse of a principal minor matrix. Several techniques are described and references to others are given.

Chapter 17 (47 pages) is devoted to the study of the errors in linear computations, the details of which are too lengthy to mention in this review. A theorem of Satterthwaite is found useful in discussing error control and estimating the accuracy of a solution.

Chapter 18 gives a summary of specific applications of these computational techniques to statistical computations that arise in the analysis of variance, and in multiple correlation and regression problems.

A final chapter is devoted to nonlinear problems and interpolation. The book is aimed at the research worker who has a desk calculator and a linear computational problem involving up to 15 or 20 variables, but only a limited background in algebra. Matrices are described and used as a computational shorthand, rather than as elements in an associative matrix algebra.

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● KINGZETT'S CHEMICAL ENCYCLOPAEDIA

Edited by *Ralph K. Strong*, Professor of Chemistry, Rose Polytechnic Institute, Terre Haute, Indiana. Eighth edition. D. Van Nostrand Co., Inc., New York, 1952. xii + 1186 pp. 15 × 23 cm. $16.

THOSE who wonder what function is served by a chemical encyclopedia may be interested in one paragraph from the foreword by Sir Robert Robinson:

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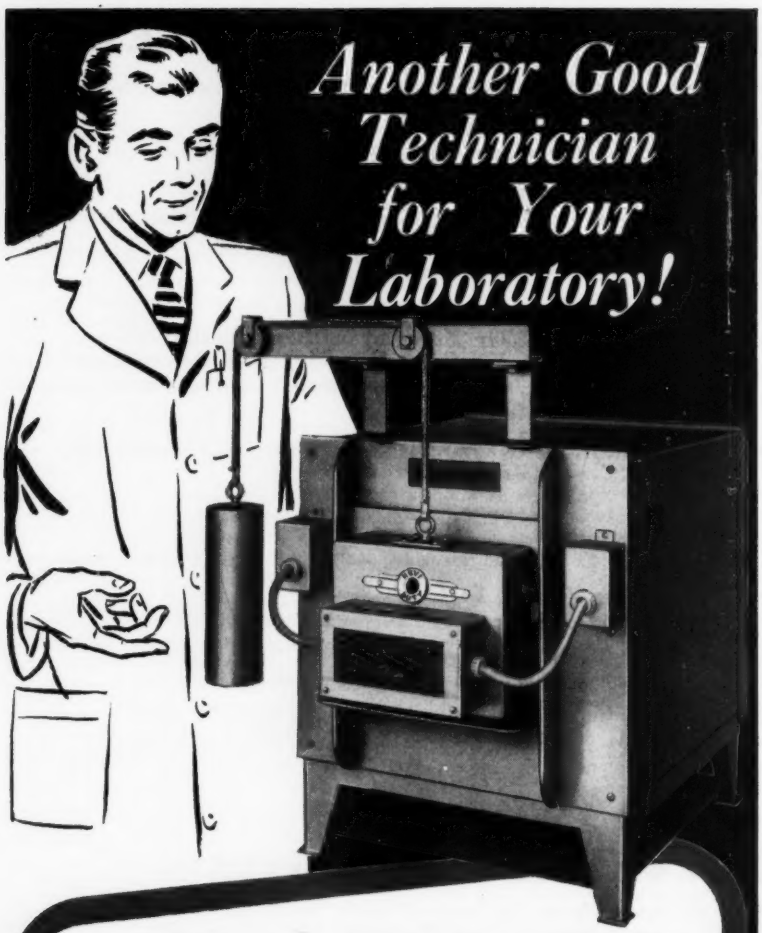

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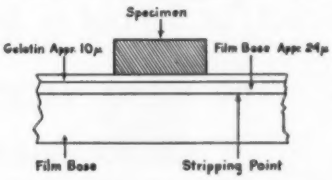

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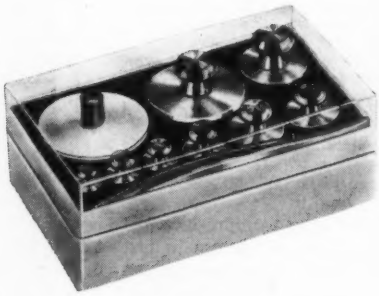

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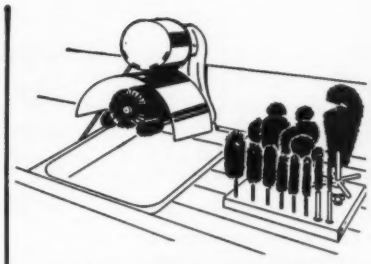

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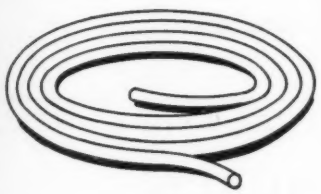

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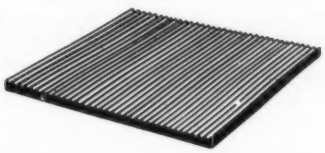

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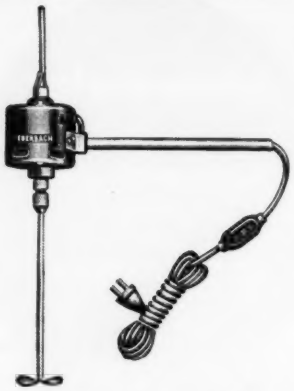

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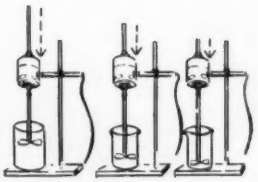

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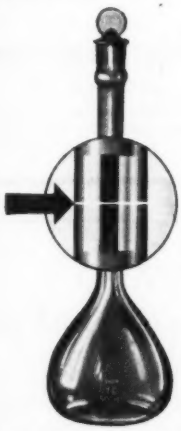

Complete details regarding the new "Emson" Volumetric Flasks are available on request, from E. Machlett & Son, 220 E. 23rd St., New York 10, New York.

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Complete description and details are available in Product Bulletin A-1 from The Perkin-Elmer Corp., Norwalk, Connecticut.

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● Bios Laboratories, Inc., 17 W. 60th St., New York 23, New York, announces the availability of the following new chemicals: Ceryl Alcohol; Chelidonic Acid (Jervasic Acid), 4-oxo-1,4-Pyrah-2,6-Dicarboxylic Acid; 2,4-Dichloropyrimidine; Myoglobin; Quinic Acid, (Hexahydro-1,3,4,5 - Tetrahydroxybenzoic Acid);

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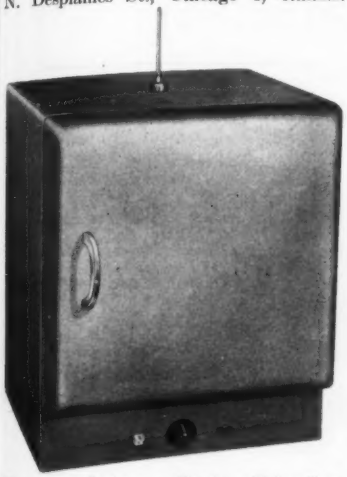

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● E. H. Sargent & Co. has just published the *Centennial Anniversary Edition* of their catalogue. This 1500-page volume illustrates and describes their complete line of scientific instruments, apparatus, and chemicals. Copies may be obtained by writing E. H. Sargent & Co., 4647 W. Foster Ave., Chicago 30, Illinois.

● A folder on the *Harshaw Crystal Model Kit* is now available from Harshaw Scientific, Division of the Harshaw Chemical Co., 1945 E. 97th St., Cleveland 6, Ohio. The folder illustrates and describes this kit which was developed to fill the need for a simple and effective means of visualizing, through a demonstration model, various crystalline structures.

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● A new 48-page booklet on *Constant Temperature Equipment* is announced by Central Scientific Co. It contains useful information about ovens, incubators, water heaters, and thermoregulators and includes details of their construction and operation. A copy may be had without charge by writing Central Scientific Co., 1700 Irving Park Rd., Chicago 13, Illinois. Ask for Bulletin No. 5B.

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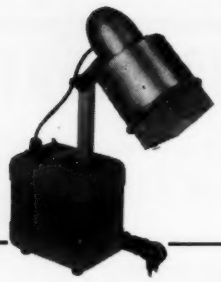

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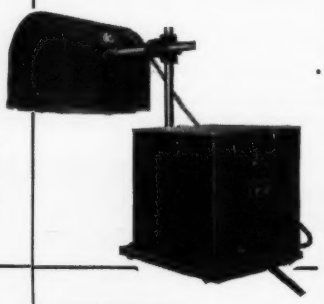

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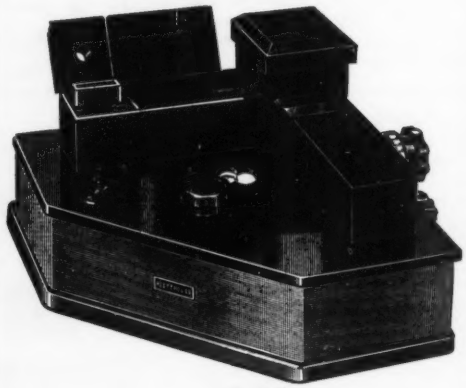

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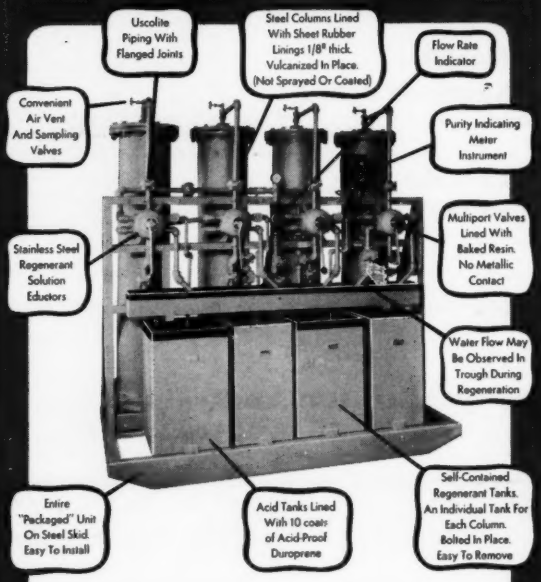
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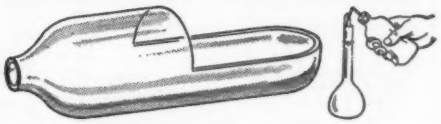

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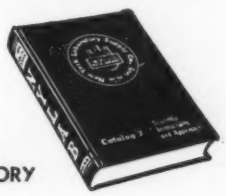

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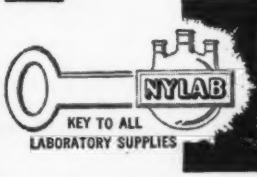

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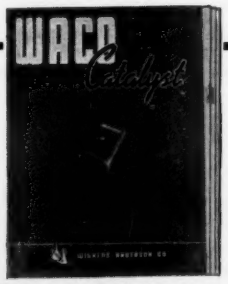

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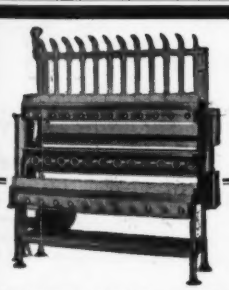

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